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L10: Entry 2 of 12

File: USPT

May 15, 2001

US-PAT-NO: 6231343

DOCUMENT-IDENTIFIER: US 6231343 B1

TITLE: Method for bleaching discolored tooth by titanium dioxide photocatalyst

DATE-ISSUED: May 15, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
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US-CL-CURRENT: 433/215; 433/216

CLAIMS:

What is claimed is:

1. A method for bleaching discolored teeth comprising applying a solution or paste or both comprising a titanium dioxide powder and hydrogen peroxide solution onto the surface of the discolored teeth, and irradiating the discolored teeth with light, thereby bleaching the teeth by resultant photocatalytic action.
2. The method for bleaching discolored teeth according to claim 1, wherein said irradiating light is violet or blue visible light.
3. The method for bleaching discolored teeth according to claim 1, which comprises:
 - a) cleaning surfaces of the discolored teeth;
 - b) moisture-proofing said cleaned surfaces; and
 - c) coating said moisture-proofed surfaces with said solution or paste or both of said titanium dioxide powder and said hydrogen peroxide, and irradiating said coated surfaces with visible light.
4. The method for bleaching discolored teeth according to claim 3, wherein step c) is repeated, whereby fresh paste or solution or both is applied and irradiated every 15 to 20 minutes.
5. The method for bleaching discolored teeth according to claim 4, wherein step c) is repeated 2 or 3 times.
6. The method for bleaching discolored teeth according to claim 5, wherein step c) is repeated 4 or 5 times.
7. The method for bleaching discolored teeth according to claim 1, wherein said solution or past further comprises trace amounts of iron salts.
8. A bleaching composition for bleaching discolored teeth by photocatalytic

action produced by irradiation with light, wherein

said bleaching composition comprises as active ingredients a combination of 6% or less hydrogen peroxide solution and crystalline titanium dioxide photocatalyst, wherein

said photocatalyst produces the photocatalytic action and reacts with the hydrogen peroxide to produce active oxygen when irradiated with light.

9. The bleaching composition according to claim 8, which consists essentially of a solution/paste of titanium dioxide with a particle diameter of about 5 to 60 nm and 3% or less hydrogen peroxide solution.

10. The bleaching composition according to claim 8, which further comprises trace amounts of iron salts.

11. A method for producing a bleaching composition for bleaching discolored teeth by photocatalytic action produced by irradiation with light, which method comprises:

blending 6% or less hydrogen peroxide solution with a crystalline titanium dioxide photocatalyst powder, wherein

said photocatalyst producing the photocatalytic action and reacts with hydrogen peroxide to produce active oxygen when irradiated with light.

12. The method for producing a bleaching composition according to claim 11, wherein 3% or less hydrogen peroxide solution is blended with the crystalline titanium dioxide photocatalyst powder.

13. The method for producing a bleaching agent according to claim 11, wherein the crystalline titanium dioxide photocatalyst powder is anatase-type titanium dioxide.

14. A system for bleaching teeth, comprising a combination of a bleaching composition, materials or device for applying said bleaching composition, irradiating device, and optionally other dental treatment materials, wherein the bleaching composition comprises as active ingredients a combination of 6% or less hydrogen peroxide solution and crystalline titanium dioxide photocatalyst,

said photocatalyst producing the photocatalytic action and reacting with the hydrogen peroxide to produce active oxygen when irradiated with light.

15. The system for bleaching teeth according to claim 14, wherein the radiating device generates visible violet light.

16. The system for bleaching teeth according to claim 15, wherein the radiating device generates LED violet light.

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L10: Entry 2 of 12

File: USPT

May 15, 2001

DOCUMENT-IDENTIFIER: US 6231343 B1

TITLE: Method for bleaching discolored tooth by titanium dioxide photocatalyst

Abstract Text (1):

The present invention relates to a method for bleaching a discolored tooth, comprising the steps of applying a solution/paste of titanium dioxide powder and hydrogen peroxide solution onto the surface of discolored teeth, and bleaching the tooth based on the photocatalytic action that is produced by irradiating this area with light; and to a bleaching agent for applying onto the surface of discolored tooth to bleach the discolored tooth based on the photocatalytic action that is produced by irradiating this area with light, the bleaching agent comprising as active ingredients a combination of hydrogen peroxide solution and titanium dioxide producing photocatalytic action when irradiated with light.

Brief Summary Text (3):

The present invention relates to the utilization of photocatalytic action to bleach and remove colored sediment on (stained or discolored) teeth, and more particularly to a method for bleaching discolored teeth comprising the steps of applying a bleaching agent comprising a specific composition having photocatalytic action on the surface of discolored teeth, and bleaching the teeth based on the photocatalytic action that is produced by irradiating this area with light; a novel bleaching agent comprising a solution/paste of 3% or less aqueous hydrogen peroxide and titanium dioxide producing photocatalytic action when irradiated with light, which is useful for such bleaching; a method for producing such a bleaching agent; and a bleaching system comprising a combination of the aforementioned bleaching agent and the like.

Brief Summary Text (9):

1) Bleaching which features the use of 30% H.sub.2 O.sub.2 as the chemical agent, and the combined use of light and heat on the aqueous hydrogen peroxide.

Brief Summary Text (11):

2) Bleaching which features the use of 30% H.sub.2 O.sub.2 as the chemical agent, and the concurrent use of high frequency current on the aqueous hydrogen peroxide.

Brief Summary Text (13):

3) Bleaching in which a paste comprising Aerosil (silica fine powder) mixed with 35% H.sub.2 O.sub.2 is used as the chemical agent, and the aqueous hydrogen peroxide and Aerosil paste is applied.

Brief Summary Text (21):

Many other bleaching methods have also been reported, such as the dental bleaching agent and method comprising a mixture of aqueous hydrogen peroxide and ortho-phosphoric acid (Japanese Laid-Open Patent Application H8-143436/1996), the bleaching agent comprising a mixture of silicic anhydride with aqueous hydrogen peroxide, and the vital bleaching method comprising the application of such a bleaching agent (Japanese Laid-Open Patent Application H5-320033/1993), and the dental bleaching composition comprising a dental bleaching agent (such as hydrogen-urea peroxide, hydrogen peroxide-carbamide, and carbamide peroxide) and a matrix material (such as carboxymethylene), and a method for bleaching teeth using the above (Japanese Laid-Open Patent Application H8-113520/1996).

Brief Summary Text (30):

In conventional bleaching methods, however, the primary chemical agent is 30 to 35%

aqueous hydrogen peroxide, which is highly corrosive, and the oxidative action of which is the basis of bleaching.

Brief Summary Text (31):

All of the various bleaching methods currently used in Japan can be said to comprise, as noted above, a combination of 30 to 35% aqueous hydrogen peroxide, various utensils, and other chemical agents. There is an example of a bleaching method employed in the United States which features the use of 10% urea peroxide instead of 30 to 35% aqueous hydrogen peroxide, but this method is currently embroiled in controversy over problems in terms of efficacy and safety, and has yet to gain approval in Japan.

Brief Summary Text (32):

Bleaching methods covered as insured treatment are currently limited to pulpless teeth, and in some cases pulpectomy is performed on non-carries teeth for the purpose of bleaching. In addition, the use of highly toxic 30 to 35% aqueous hydrogen peroxide in various bleaching methods restricts operations and the like in a variety of ways, while limited bleaching effects for pulped teeth in particular have been indicated.

Brief Summary Text (36):

The present invention relates to a method for bleaching discolored teeth, comprising the steps of applying a solution/paste of titanium dioxide powder and aqueous hydrogen peroxide to the surface of discolored teeth, and bleaching the teeth based on the photocatalytic action that is produced by irradiating this area with light; and also to a bleaching agent which is applied to the surface of discolored teeth to bleach the discolored teeth based on the photocatalytic action that is produced by irradiating this area with light, the aforementioned bleaching agent comprising as active ingredients a combination of aqueous hydrogen peroxide and titanium dioxide producing photocatalytic action when irradiated with light.

Brief Summary Text (39):

As a result of extensive research in light of the foregoing to establish a novel bleaching method that would be safer and simpler, and that would also afford better bleaching effects, the inventors perfected the present invention upon discovering that the intended objectives can be achieved by the combined use of active ingredients comprising aqueous hydrogen peroxide and titanium dioxide having photocatalytic action.

Brief Summary Text (43):

(1) A method for bleaching a discolored tooth, comprising the steps of applying a solution/paste of a titanium dioxide powder and hydrogen peroxide solution onto the surface of the discolored tooth, and irradiating this area with light to bleach the tooth based on the resultant photocatalytic action.

Brief Summary Text (45):

(3) A bleaching agent for applying onto a surface of a discolored tooth to bleach the discolored tooth based on a photocatalytic action that is produced by irradiating this area with light, said bleaching agent comprising as active ingredients a combination of hydrogen peroxide solution and titanium dioxide which produces the photocatalytic action when irradiated with light.

Brief Summary Text (46):

(4) The bleaching agent according to (3) above, consisting of a solution/paste of titanium dioxide with a particle diameter of about 5 to 60 nm and 3% or less hydrogen peroxide solution.

Brief Summary Text (47):

(5) A method for producing a bleaching agent for applying onto a surface of a discolored tooth to bleach the discolored teeth based on the photocatalytic action that is produced by irradiating this area with light, said method for producing a bleaching agent comprising the step of blending hydrogen peroxide solution with a titanium dioxide powder producing photocatalytic action when irradiated with light.

Brief Summary Text (48):

(6) The method for producing a bleaching agent according to (5) above, comprising blending 3% or less hydrogen peroxide solution with a titanium dioxide powder producing photocatalytic action when irradiated with light.

Brief Summary Text (49):

(7) The method for producing a bleaching agent according to (5) or (6) above, wherein anatase-type titanium dioxide is arranged as the titanium dioxide powder.

Brief Summary Text (54):

The chemical agents and utensils used in the present invention may essentially comprise titanium oxide powder, aqueous hydrogen peroxide, and visible light rays (radiating instrument), affording considerable stability, ease of operation, and bleaching effects. The aforementioned aqueous hydrogen peroxide is more suitably used in lower concentrations, such as a concentration of 6% or less, and preferably a lower concentration of 3% or less.

Brief Summary Text (55):

In a preferred embodiment, the bleaching agent of the present invention is composed of a solution/paste of titanium dioxide powder and aqueous hydrogen peroxide in a low concentration of, for example, 6% or less (and preferably 3% or less). Examples of desirable titanium dioxide include, but are not limited to, titanium dioxide having a particle diameter of about 5 to 60 nm, and particularly anatase type titanium dioxide fine particles or materials based thereon. Any titanium dioxide having similar effects, that is, producing photocatalytic action, can be used, no matter what the shape or properties. Advantages of a smaller particle diameter in this case are that higher activity can be anticipated, good effects can be obtained with lower amounts, lower amounts can be used, high bleaching effects can be obtained more rapidly because thinner coating films can be used, for example, and so forth.

Brief Summary Text (56):

The weight ratio of the aforementioned ingredients can be adjusted as desired depending, for example, on whether the degree of discoloration is lighter or greater, so that products suited to individual cases can be prepared. The bleaching agent of the present invention is ordinarily, but not only, used in the form of a uniform transparent solution or paste by blending, kneading, and dispersing the titanium dioxide powder in aqueous hydrogen peroxide of low concentration. Any type that is prepared in a manner similar to this is encompassed within the range of the present invention. The term "solution/paste" as used in the present invention is defined as any having the aforementioned meaning. In this case, the means and devices for preparing the bleaching agent such as by blending, kneading, dispersion or otherwise manipulating the aforementioned ingredients, as well as the means and the like for applying the bleaching agent, are not particularly limited. Any suitable type can be used. An example of a desirable method for applying the bleaching agent to dental surfaces is to directly coat the dental surface with the bleaching agent.

Brief Summary Text (57):

At this time, fabric, paper, glass cloth, ceramic paper, an organic gel, an inorganic gel, or the like can be impregnated with the bleaching agent of the present invention, that is, a solution or paste of the aqueous hydrogen peroxide and titanium dioxide having photocatalytic action, and the impregnated product can be applied to the dental surface and irradiated with light. Other suitable methods and means can also be used, such as a method in which the aforementioned bleaching agent is supported on a suitable carrier, and is fitted or applied to a tooth or row of teeth.

Brief Summary Text (59):

Discolored teeth can be bleached with the aforementioned bleaching agent by repeatedly applying a solution or paste of a titanium dioxide powder and 3% or less aqueous hydrogen peroxide to a dental surface, for example, and irradiating the coated area. The number of times the material is applied and irradiated may be suitably adjusted according to how light or heavy the staining is. The aforementioned solution or paste should usually be applied by coating or the like using fresh solution or paste about every 15 to 20 minutes. The interval and

frequency should be suitably determined according to the state of the teeth.

Brief Summary Text (61):

The primary action of the bleaching agent in the present invention is bleaching action based on the synergistic action of the titanium dioxide photocatalyst and low concentration aqueous hydrogen peroxide (for example, 6% or less, and preferably 3% or less, aqueous hydrogen peroxide).

Brief Summary Text (62):

When the titanium dioxide photocatalyst is irradiated with light, electrons and positive holes are produced, reacting with hydrogen peroxide to produce active oxygen. The active oxygen has far greater oxidizing power than ozone, and can oxidize nearly all organic materials into carbon dioxide. Even when n type semiconductor titanium powder with a relatively substantial band gap is used in the form of a solution with 3% aqueous hydrogen peroxide, for example, light radiation readily results in the production of active oxygen having potent oxidizing power, ensuring higher levels of charge separation, electron hole mobility, reactivity with protons or hydroxyl groups, or the like than when used alone, so that synergistic action is produced in addition to the oxidizing action of the 3% aqueous hydrogen peroxide itself.

Brief Summary Text (87):

A solution of titanium oxide and, for example, 6% or less, and preferably 3% or less, aqueous hydrogen peroxide penetrates between the enamel prisms and dentine, and bleaching is brought about as colored substances are degraded by the oxidative and reductive action of the photocatalyst. The bleaching method of the present invention affords high bleaching effects for discolored teeth caused by both intrinsic and extrinsic factors.

Detailed Description Text (3):

0.3 g of anatase type TiO_2 powder with a particle diameter of 7 nm was blended with 1 mL each of a) 0.5% aqueous hydrogen peroxide, b) 1.5% aqueous hydrogen peroxide, and c) 3% aqueous hydrogen peroxide, and the ingredients were kneaded and dispersed to prepare solutions of titanium dioxide and aqueous hydrogen peroxide. 1 g of anatase type TiO_2 powder with a particle diameter of 7 nm was similarly used to prepare a d) paste of 3% aqueous hydrogen peroxide. The products were then housed in containers shielded from light, giving bleaching agents.

Detailed Description Text (11):

4) The dental surface was coated with a solution of titanium oxide and 3% aqueous hydrogen peroxide, and irradiated with visible light.

Detailed Description Text (16):

Since the bleaching agent of the present invention provides excellent bleaching effects through the synergism between the bleaching action based on the titanium oxide photocatalyst and the bleaching action of the aqueous hydrogen peroxide, there are no operational restrictions such as those in the case of conventional, highly toxic 30 to 35% aqueous hydrogen peroxide. It may also be understood that the bleaching agent of the present invention can be used for both pulped and pulpless teeth, since it is highly safe.

Detailed Description Text (17):

Compared, in terms of oxidizing energy, to conventional bleaching agents based on aqueous hydrogen peroxide, the bleaching agent in the present invention can be seen to have rapidly resulted in about 2.9 times or more greater bleaching effects.

Detailed Description Text (18):

It is also evident from Table 1 that lower aqueous hydrogen peroxide concentrations tended to require a longer period of time.

Detailed Description Text (38):

(3) Concentration of Aqueous Hydrogen Peroxide

Detailed Description Text (39):

The concentration of aqueous hydrogen peroxide was varied within the range from 0.1%

to 35%, and attempts were made to bleach discolored teeth by applying and irradiating (visible light) the material in the same manner as above.

Detailed Description Text (50) :

As described above, the present invention relates to a bleaching agent for bleaching discolored teeth based on the photocatalytic action that is produced when the agent is applied to the surface of discolored teeth and is irradiated, comprising as active ingredients a combination of aqueous hydrogen peroxide and titanium dioxide producing photocatalytic action when irradiated. The present invention is extremely useful for cosmetic improvements of teeth because of the following effects: (1) the ability to provide a novel bleaching agent for discolored teeth; (2) the ability to bleach both vital teeth and pulpless teeth; (3) higher safety, better workability, and rapid bleaching effects; (4) alleviation of mental distress on treated patients; (5) the ability to control the level of color adaptation; and (6) the ability to select improvements in color adaptation desired by the treated patient.

CLAIMS:

1. A method for bleaching discolored teeth comprising applying a solution or paste or both comprising a titanium dioxide powder and hydrogen peroxide solution onto the surface of the discolored teeth, and irradiating the discolored teeth with light, thereby bleaching the teeth by resultant photocatalytic action.
3. The method for bleaching discolored teeth according to claim 1, which comprises:
 - a) cleaning surfaces of the discolored teeth;
 - b) moisture-proofing said cleaned surfaces; and
 - c) coating said moisture-proofed surfaces with said solution or paste or both of said titanium dioxide powder and said hydrogen peroxide, and irradiating said coated surfaces with visible light.
8. A bleaching composition for bleaching discolored teeth by photocatalytic action produced by irradiation with light, wherein

said bleaching composition comprises as active ingredients a combination of 6% or less hydrogen peroxide solution and crystalline titanium dioxide photocatalyst, wherein

said photocatalyst produces the photocatalytic action and reacts with the hydrogen peroxide to produce active oxygen when irradiated with light.
9. The bleaching composition according to claim 8, which consists essentially of a solution/paste of titanium dioxide with a particle diameter of about 5 to 60 nm and 3% or less hydrogen peroxide solution.
11. A method for producing a bleaching composition for bleaching discolored teeth by photocatalytic action produced by irradiation with light, which method comprises:

blending 6% or less hydrogen peroxide solution with a crystalline titanium dioxide photocatalyst powder, wherein

said photocatalyst producing the photocatalytic action and reacts with hydrogen peroxide to produce active oxygen when irradiated with light.
12. The method for producing a bleaching composition according to claim 11, wherein 3% or less hydrogen peroxide solution is blended with the crystalline titanium dioxide photocatalyst powder.
13. The method for producing a bleaching agent according to claim 11, wherein the crystalline titanium dioxide photocatalyst powder is anatase-type titanium dioxide.
14. A system for bleaching teeth, comprising a combination of a bleaching composition, materials or device for applying said bleaching composition,

irradiating device, and optionally other dental treatment materials, wherein the bleaching composition comprises as active ingredients a combination of 6% or less hydrogen peroxide solution and crystalline titanium dioxide photocatalyst,

said photocatalyst producing the photocatalytic action and reacting with the hydrogen peroxide to produce active oxygen when irradiated with light.

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May 15, 2001

US-PAT-NO: 6231343

DOCUMENT-IDENTIFIER: US 6231343 B1

TITLE: Method for bleaching discolored tooth by titanium dioxide photocatalyst

DATE-ISSUED: May 15, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
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Taoda; Hiroshi	Nagoya-shi			JP
Nonami; Toru	Nagoya-shi			JP

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APPL-NO: 09/ 508109 [PALM]

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US-CL-CURRENT: 433/215; 433/216

FIELD-OF-SEARCH: 433/215, 433/216, 433/229, 433/80

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

Search Selected

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	PAT-NO	ISSUE-DATE	PATENTEE-NAME	US-CL
<input type="checkbox"/>	<u>5766574</u>	June 1998	Christina-Beck et al.	424/53
<input type="checkbox"/>	<u>5902568</u>	May 1999	Ryles et al.	424/53
<input type="checkbox"/>	<u>5914305</u>	June 1999	Madison et al.	510/367

ART-UNIT: 372

PRIMARY-EXAMINER: Lucchesi; Nicholas D.

ABSTRACT:

The present invention relates to a method for bleaching a discolored tooth, comprising the steps of applying a solution/paste of titanium dioxide powder and hydrogen peroxide solution onto the surface of discolored teeth, and bleaching the tooth based on the photocatalytic action that is produced by irradiating this area with light; and to a bleaching agent for applying onto the surface of discolored tooth to bleach the discolored tooth based on the photocatalytic action that is produced by irradiating this area with light, the bleaching agent comprising as active ingredients a combination of hydrogen peroxide solution and titanium dioxide producing photocatalytic action when irradiated with light.

16 Claims, 0 Drawing figures

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L10: Entry 5 of 12

File: USPT

Jun 2, 1998

US-PAT-NO: 5759251

DOCUMENT-IDENTIFIER: US 5759251 A

TITLE: Titanium dioxide ceramic paint and methods of producing same

DATE-ISSUED: June 2, 1998

INVENTOR-INFORMATION:

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Mori; Kazuhiko	Tokyo			JP
Tanaka; Masanobu	Tokyo			JP
Nishizawa; Yoshihiko	Tokyo			JP

US-CL-CURRENT: 106/286.4; 106/287.19, 106/816

CLAIMS:

What we claimed is:

1. An aqueous titanium dioxide ceramic paint for forming a paint coating having excellent hydrophilicity, photocatalytic activity and light-transmittance, comprising:

(A) at least one member selected from the group consisting of orthotitanic acid, titanium (IV) ions and peroxotitanic acid, and

(B) crystalline titanium dioxide colloidal particles having an average particle size of 0.001 to 0.2 .mu.m.

the weight ratio, in terms of titanium, of the component (A) to the component (B) being from 1:0.1 to 1:200, and the ceramic paint being substantially free from impurity ions.

2. The aqueous titanium dioxide ceramic paint as claimed in claim 1, wherein the average particle size of the crystalline titanium dioxide particles is from 0.002 to 0.1 .mu.m.

3. A method of producing the aqueous titanium dioxide ceramic paint as claimed in claim 1, comprising subjecting an aqueous solution of a titanium salt to at least one treatment selected from the group consisting of:

(a) a dialysis treatment using a semipermeable membrane,

(b) an electrodialysis treatment using a semipermeable membrane, and

(c) an ion-exchange treatment using an ion-exchanger

to thereby hydrolyze and convert at least a portion of the titanium salt to be orthotitanic acid and the crystalline titanium dioxide colloidal particles, while removing impurity ions from the aqueous titanium salt solution.

4. The method as claimed in claim 3, wherein the treatments (a), (b) and (c) are carried out at a temperature of 0.degree. to 80.degree. C.

5. A method as claimed in claim 4, wherein the aqueous solution of the titanium salt is heated at a temperature of 50.degree. C. or more but less than 100.degree. C., and then, subjected to the at least one treatment selected from the treatments (a), (b) and (c).

6. The method as claimed in claim 3 or 5, wherein the aqueous titanium salt solution is an aqueous solution containing at least one member selected from the group consisting of titanium oxychloride and titanium oxysulfate.

7. The method as claimed in claim 3, wherein the aqueous titanium salt solution is mixed with at least one member selected from the group consisting of alkali metal hydroxides and ammonia in an amount of less than 4 moles per mole of titanium present in the aqueous titanium salt solution, and the resultant mixed aqueous solution is heated at a temperature of 50.degree. C. or more but less than 100.degree. C., and then subjected to the at least one treatment selected from the treatments (a), (b) and (c).

8. A method of producing the titanium dioxide ceramic paint as claimed in claim 1, comprising the steps of:

mixing an aqueous solution of a titanium salt with at least one member selected from the group consisting of alkali metal hydroxides and ammonia in an amount of less than 4 moles per mole of titanium present in the aqueous titanium salt solution; and

heating the resultant mixed aqueous solution at a temperature of 50.degree. C. or more but less than 100.degree. C., to thereby hydrolyze and convert at least a portion of the titanium salt to the orthotitanic acid and the crystalline titanium dioxide colloidal particles; and

mixing the resultant titanium dioxide colloidal particle-containing aqueous solution with at least one member selected from the group consisting of alkali metal hydroxides and ammonia to cause a precipitate to be formed;

collecting the resultant precipitate from the aqueous solution; and

dispersing the collected precipitate in a dispersing medium consisting of a member selected from the group consisting of water, aqueous solutions of peroxide compounds and aqueous solutions of complexing agents for titanium .

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File: USPT

Jun 2, 1998

DOCUMENT-IDENTIFIER: US 5759251 A

TITLE: Titanium dioxide ceramic paint and methods of producing same

Brief Summary Text (35):

The orthotitanic acid usable for the present invention exhibits a high solubility in acid aqueous solutions and substantially no crystallizability and thus can be definitely distinguished from the crystalline titanium dioxide particles. Also, the titanium dioxide particles usable for the present invention include metatitanic acid, anatase titanium dioxide, and rutile titanium dioxide particles.

Brief Summary Text (37):

The peroxotitanic acid usable for the ceramic paint of the present invention can be prepared in the state of an aqueous solution by adding an aqueous hydrogen peroxide solution to an aqueous solution of orthotitanic acid prepared by the above-mentioned procedures.

Brief Summary Text (39):

The titanium dioxide ceramic paint of the present invention must comprise the crystalline titanium dioxide particles in addition to the orthotitanic acid, titanium (IV) ions and/or peroxotitanic acid. The crystal form of the titanium dioxide particles is most preferably anatase and second most preferably rutile.

Brief Summary Text (45):

Also, the peroxotitanic acid and titanium dioxide particle-containing aqueous paint of the present invention can be prepared by adding a peroxide compound, for example, hydrogen peroxide, into the aqueous orthotitanic salt solution, before or after the hydrolysis procedures. In this case, the content of peroxotitanic acid in the resultant aqueous paint can be adjusted to 0 to 100% by controlling the amount of the peroxide compound to be added to the aqueous titanium salt solution. Namely, when no peroxide compound is added, the resultant aqueous paint contains orthotitanic acid and is free from peroxotitanic acid. Also, when the peroxide compound is added in an amount equimolar or more to the molar amount of the orthotitanic acid, the resultant aqueous paint contains peroxotitanic acid and is free from orthotitanic acid.

Brief Summary Text (51):

When anhydrous titanium chloride is used as a starting material, an aqueous titanium chloride solution can be prepared by gradually dissolving anhydrous titanium chloride in pure water while cooling with ice. Also, when titanium trichloride is employed as a starting material, first, titanium (III) ions are oxidized into titanium (IV) ions with an oxidizing agent, for example, hydrogen peroxide, and second, the resultant titanium tetrachloride is subjected to the aqueous paint-producing procedures.

Brief Summary Text (98):

In the dispersion of the collected precipitate, the peroxide compound is preferably selected from hydrogen peroxide, sodium peroxide, and barium peroxide. The peroxide compound react with at least a portion of orthotitanic acid and titanium (IV) ions to produce peroxotitanic acid. When hydrogen peroxide is used, the addition of hydrogen peroxide can be carried out before the collection of the precipitate. Also when sodium peroxide or barium peroxide is used, the resultant aqueous paint may be further subjected to the above-mentioned treatment (a), (b) and/or (c) to remove sodium ions or barium ions from the aqueous paint.

Brief Summary Text (100):

The aqueous titanium dioxide ceramic paint produced by the method (1) or (2) of the present invention contains a large content of crystalline titanium dioxide particles in addition to orthotitanic acid, titanium (IV) ions and/or peroxotitanic acid. Usually, the titanium dioxide particles have an anatase crystal form. However, depending on the conditions of the heating treatment, the titanium dioxide particles may include rutile crystal particles.

Brief Summary Text (104):

The aqueous titanium dioxide ceramic paint of the present invention is employed by coating the aqueous ceramic paint on a surface of a substrate article, drying the coated paint layer, and baking the dried paint layer at a temperature of, preferably 100.degree. to 700.degree. C., more preferably 200.degree. to 500.degree. C. Usually, the resultant baked paint coating contains anatase titanium dioxide crystals. When baked at a high temperature of 700.degree. C. or more, however, at least a portion of the anatase crystals may be converted to rutile crystals. The crystal conversion does not cause the hydrophilicity of the paint coating to be reduced or be lost.

Detailed Description Text (4):

In each of Examples 1 to 4, an aqueous paint was prepared by mixing an aqueous 10% sodium hydroxide solution to an aqueous 5% titanium tetrachloride solution to cause a precipitate to generate, collecting the precipitate consisting of orthotitanic acid by filtration, rinsing the collected precipitate with water, dispersing the precipitate in water, mixing the resultant orthotitanic acid-containing aqueous liquid with crystalline titanium dioxide particles, and then mixing the resultant liquid with the complexing agent in the amount as indicated in Table 1 in each of Examples 1, 3 and 4, and with the aqueous hydrogen peroxide solution in the amount sufficient to realize the concentration of peroxotitanic acid as shown in Table 1 in Example 2, and fully agitating the resultant mixture liquid by using a homomixer. The above-mentioned crystalline titanium dioxide particles were available under the trademark of titanium dioxide P-25 (anatase or rutile) made from Nihon Aerosil K.K. The above-mentioned complexing agent consisted of acetylacetone in Example 1, lactic acid in Example 3 and oxalic acid in Example 4.

Detailed Description Text (7):

An aqueous 10% titanium tetrachloride solution was mixed with sodium hydroxide in an amount of 1 mole per mole of titanium present in the titanium tetrachloride solution, the mixed liquid was heated at a temperature of 90.degree. C. for 40 minutes, the heated liquid was mixed with an aqueous 5% sodium hydroxide solution in such an amount that the resultant mixed liquid became neutral, to cause a precipitate to form, the resultant precipitate comprising orthotitanic acid and titanium dioxide was dispersed in water, the resultant aqueous dispersion was mixed with an aqueous hydrogen peroxide solution in such an amount that the resultant mixed liquid had the concentration of peroxotitanic acid as shown in Table 1, the resultant mixture liquid was fully agitated by using a homomixer. As aqueous titanium dioxide ceramic paint was obtained.

Detailed Description Text (8):

As a result of analysis, it was confirmed that the resultant aqueous paint contained anatase crystalline titanium dioxide particles having an average particle size of 0.08 .mu.m.

Detailed Description Text (11):

An aqueous 10% titanium tetrachloride solution was mixed with sodium hydroxide in an amount of 2 moles per mole of titanium present in the titanium tetrachloride solution, the mixed liquid was heated at a temperature of 75.degree. C. for 20 minutes, the heated liquid was mixed with an aqueous 5% sodium hydroxide solution in such an amount that the resultant mixed liquid became neutral, to cause a precipitate to generate, the resultant precipitate comprising orthotitanic acid and titanium dioxide was dispersed in water, the resultant aqueous dispersion was mixed with, in Example 7, gluconic acid in the concentration shown in Table 1, and in Example 8, an aqueous hydrogen peroxide solution in such an amount that the resultant mixed liquid had the concentration of peroxotitanic acid as shown in Table

1, the resultant mixture liquid was fully agitated by using a homomixer. As aqueous titanium dioxide ceramic paint was obtained.

Detailed Description Text (12):

As a result of analysis, it was confirmed that the resultant aqueous paint contained anatase crystalline titanium dioxide colloidal particles having an average particle size of 0.05 .mu.m.

Detailed Description Text (20):

An aqueous 20% by weight titanium trichloride solution was placed in a beaker and diluted with water. The diluted solution was mixed with an aqueous 31% hydrogen peroxide solution until violet-colored titanium (III) ions present in the aqueous solution were completely converted to colorless titanium (IV) ions. The resultant aqueous titanium tetrachloride solution was mixed with an aqueous solution of 10% by weight of sodium hydroxide in an amount of 2 moles per mole of titanium present in the aqueous solution. The mixed liquid was heated at a temperature of 70.degree. C. for 30 minutes and then cooled with water to a temperature of 30.degree. C. The resultant liquid was subjected to a diffusion dialysis treatment using an RO membrane at a temperature of 40.degree. C. while passing deionized water through the membrane, to prepare an aqueous titanium dioxide ceramic paint substantially free from impurity ions.

Detailed Description Text (22):

An aqueous 20% by weight titanium trichloride solution was placed in a beaker and diluted with water. The diluted solution was mixed with an aqueous 31% hydrogen peroxide solution until violet-colored titanium (III) ions present in the aqueous solution were completely converted to colorless titanium (IV) ions. The resultant aqueous titanium tetrachloride solution was mixed with an aqueous solution of 10% by weight of sodium hydroxide in an amount of 0.24 mole per mole of titanium present in the aqueous solution. The mixed liquid was heated at a temperature of 95.degree. C. for 5 minutes and then cooled with water to a temperature of 30.degree. C. The resultant liquid was subjected to a diffusion dialysis treatment using anion exchange and cation exchange membranes at a temperature of 30.degree. C. while passing deionized water through the membrane, to prepare an aqueous titanium dioxide-containing liquid substantially free from impurity ions. The aqueous titanium dioxide-containing liquid was mixed with a complexing agent consisting of acetylacetone in an amount of 1.2% by weight.

Detailed Description Text (55):

In each of Comparative Examples 1 to 3, an aqueous titanium dioxide ceramic paint was prepared by mixing an aqueous solution of 5% by weight of titanium tetrachloride with an aqueous solution of 10% by weight of sodium hydroxide to generate a precipitate, the precipitate was collected and rinsed with water, the resultant orthotitanic acid precipitate was dispersed in water, the resultant aqueous orthotitanic acid solution was mixed with titanium dioxide particles and then with an aqueous hydrogen peroxide solution in such an amount that the resultant solution contained peroxotitanic acid in the concentration as shown in Table 1, and the resultant mixture was fully agitated. The mixed titanium dioxide particles were, in Comparative Example 1, rutile type titanium dioxide pigment particles having an average particle size of 0.5 .mu.m, in Comparative Example 2, titanium dioxide pigment particles (anatase or rutile) (trademark: P-25, made by Nihon Aerosil K.K.) and in Comparative Example 3, anatase type titanium dioxide colloidal particles having an average particle size of 0.01 .mu.m and in the form of an aqueous colloidal solution, prepared by heating an aqueous solution of 8% by weight of titanium oxychloride at a temperature of 75.degree. C. for 20 minutes, and subjected to the heated solution to a diffusion dialysis treatment using an ion-exchange membrane.

Detailed Description Text (71):

A sample of an aqueous paint was filtered through a 5 C filter paper sheet, the resultant filtrate was acidified with hydrochloric acid and then mixed with hydrogen peroxide to convert orthotitanic acid and titanium (IV) ions to peroxotitanic acid. The absorbance of the resultant modified filtrate was measured at a wavelength of 430 nm by the same method as mentioned above. From the measurement result, the total concentration of orthotitanic acid and titanium (IV) ions in the sample was

determined. When the sample contained peroxotitanic acid, the concentration of the peroxotitanic acid in the sample was determined by the method mentioned in the above item (2), and resultant peroxotitanic acid concentration is subtracted from the total concentration of orthotitanic acid and/or titanium (IV) ions and peroxotitanic acid determined above.

CLAIMS:

1. An aqueous titanium dioxide ceramic paint for forming a paint coating having excellent hydrophilicity, photocatalytic activity and light-transmittance, comprising:

(A) at least one member selected from the group consisting of orthotitanic acid, titanium (IV) ions and peroxotitanic acid, and

(B) crystalline titanium dioxide colloidal particles having an average particle size of 0.001 to 0.2 μm .

the weight ratio, in terms of titanium, of the component (A) to the component (B) being from 1:0.1 to 1:200, and the ceramic paint being substantially free from impurity ions.

2. The aqueous titanium dioxide ceramic paint as claimed in claim 1, wherein the average particle size of the crystalline titanium dioxide particles is from 0.002 to 0.1 μm .

3. A method of producing the aqueous titanium dioxide ceramic paint as claimed in claim 1, comprising subjecting an aqueous solution of a titanium salt to at least one treatment selected from the group consisting of:

(a) a dialysis treatment using a semipermeable membrane,

(b) an electrodialysis treatment using a semipermeable membrane, and

(c) an ion-exchange treatment using an ion-exchanger

to thereby hydrolyze and convert at least a portion of the titanium salt to be orthotitanic acid and the crystalline titanium dioxide colloidal particles, while removing impurity ions from the aqueous titanium salt solution.

8. A method of producing the titanium dioxide ceramic paint as claimed in claim 1, comprising the steps of:

mixing an aqueous solution of a titanium salt with at least one member selected from the group consisting of alkali metal hydroxides and ammonia in an amount of less than 4 moles per mole of titanium present in the aqueous titanium salt solution; and

heating the resultant mixed aqueous solution at a temperature of 50.degree. C. or more but less than 100.degree. C., to thereby hydrolyze and convert at least a portion of the titanium salt to the orthotitanic acid and the crystalline titanium dioxide colloidal particles; and

mixing the resultant titanium dioxide colloidal particle-containing aqueous solution with at least one member selected from the group consisting of alkali metal hydroxides and ammonia to cause a precipitate to be formed;

collecting the resultant precipitate from the aqueous solution; and

dispersing the collected precipitate in a dispersing medium consisting of a member selected from the group consisting of water, aqueous solutions of peroxide compounds and aqueous solutions of complexing agents for titanium.

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L10: Entry 9 of 12

File: USPT

Dec 31, 1991

US-PAT-NO: 5076849

DOCUMENT-IDENTIFIER: US 5076849 A

TITLE: Transparent metal oxide pigment and method for its preparation

DATE-ISSUED: December 31, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Vapaaoksa; Pekka J.	Pori			FI
Eskelinen; Pekka J.	Pori			FI

US-CL-CURRENT: 106/481; 106/403, 106/436, 106/456, 106/489

CLAIMS:

We claim:

1. Method for the preparation of transparent metal oxide pigment-comprising dissolution of a substantial portion of the silicon dioxide from based particles coated with transparent metal oxide, characterized in that the dissolution particles whose silicon dioxide portion is substantially soluble in base and metal oxide portion- is substantially insoluble in base and carrying out the dissolution with base.

2. Method according to claim 1, characterized in that the particles are metal-oxide-coated silicate or glass particles, which have been pretreated to achieve base solubility by extraction with mineral acids selected from the group consisting of, sulphuric acid, hydrochloric acid or nitric acid, together with an oxidizer, namely hydrogen peroxide.

3. Method according to claim 2, characterized in that the silicate particles consist of layer silicate, selected from the group consisting of phlogopite or biotite.

4. Method according to claim 1, characterized in that the particles are metal-oxide-coated silicon dioxide particles or metal-oxide-coated.

5. Method according to claims 1, 2, 3 or 4, characterized in that the transparent metal oxide is titanium dioxide.

6. Method according to claims 1, 2, 3 or 4, characterized in that the silicon dioxide based particles coated with transparent metal dioxide have been prepared by precipitating the metal as hydroxide or oxihydrate onto the surface of the silicon dioxide based particles and then by thus calcinating the particles obtained.

7. Method according to claims 1, 2, 3 or 4, characterized in that the dissolution with base is carried out with sodium hydroxide or potassium hydroxide.

8. Method according to claims 1, 2, 3 or 4, characterized in that the dissolution with base is carried out with an aqueous solution the consistency of whose base is 1-30% by weight.

9. A transparent metal oxide pigment, characterized in that portion of the shape of the particles corresponds to the form of the outer surface of the silicon dioxide based particles and the particles are in the form of single closed sheets or flakes.

10. A pigment according to claim 9, characterized in that the greatest dimension of the particles is 1-300/um and the thickness is 0.001-0.3/um.

11. Pigment according to claim 9 or 10, characterized in that it does not contain more than 10% by weight of silicon dioxide.

12. Pigment according to claim 9 or 10, characterized in that it contains at least 60% by weight of titanium dioxide.

13. Pigment according to claim 9 or 10, characterized in that it contains at least 60% by weight of iron oxide.

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L10: Entry 9 of 12

File: USPT

Dec 31, 1991

DOCUMENT-IDENTIFIER: US 5076849 A

TITLE: Transparent metal oxide pigment and method for its preparation

Brief Summary Text (2):

Attempts have been made to produce a nacreous effect, interference color and color brightness by means of transparent metal oxides by stratifying them on silicon dioxide based particles suitable in size and shape. Thus, nacreous pigment has been prepared by coating layer silicate particles, such as mica particles, with a thin layer of transparent metal oxide, such as titanium dioxide in anatase or rutile form. Hereby, the problem has been that most mica-like layer silicates are too dark to form the substrate of a metal oxide pigment. The lighter mica sorts, such as muscovite, are not so common as the dark ones and, accordingly, more expensive. Moreover, the layer silicates get dark in the calcination process used in the preparation of a metal-oxide-coated mica product.

Brief Summary Text (5):

An endeavour has been made in the U.S. Pat. No. 4,192,691 to solve these problems. In the specification there is described a treatment of metal-oxide-coated muscovite particles at elevated temperature together with aqueous acid solution containing hydrofluoric acid and mineral acid until the desired amount of muscovite mica has been extracted. The substantially coreless pigment is well-dispersing and its interference color is brighter than an untreated pigment. By this known method using a very strong acid composition, it is possible to extract very efficiently mica from mica particles coated with a transparent metal oxide. A drawback is, however, that a strong acid composition also dissolves the titanium oxide. This holds good, particularly, for titanium oxide in anatase crystal form, a layer of which becomes so much thinner that the interference color of the pigment changes due to this treatment. Hereby, it is very difficult to regulate the extraction process so exactly that the desired color is achieved. Moreover, a lot of hard-treatable fluoride remain is produced in the process.

Brief Summary Text (13):

The transparent metal oxide pigment according to the invention can consist of any suitable metal oxide, such as titanium dioxide in anatase or rutile form, oxide of trivalent iron, zirconium oxide, zink dioxide, antimon oxide, chromium oxide, cobalt oxide, etc. It can also consist of a mixture of oxides, such as zinc/antimon oxide, cobalt/aluminate, etc. As the method according to the prior art is particularly detrimental to titanium dioxide in anatase crystal form, the present invention is particularly well suited for the preparation of anatase pigment.

Brief Summary Text (16):

In the embodiment of the invention in which in the beginning of the base the insoluble particles are treated with acid for achieving the base solubility, it is preferably to use mineral acid, such as sulphuric acid, hydrochloric acid or nitric acid, for the dissolution. Moreover, it is preferable to carry out the acid dissolution together with some oxidizer, such as nitric acid or hydrogen peroxide. The concentration of the acid is preferably about 0.7-70% by weight, preferably about 1-50% by weight. The dissolution in acid is carried out at a temperature below about 100.degree. C., preferably between 20.degree. and 100.degree. C., and the dissolution time is preferably about 2-30 hours.

Detailed Description Text (13):

Acid-treated, titanium dioxide-coated mica pigment is elutriated into 15 g of water.

30 g of sodium hydroxide granules are added. The mixture is heated in 80.degree. C. for 5 hours. The pigment is washed and dried. The titanium dioxide content of the product is over 90%. The titanium dioxide is in rutile form. The analysis gave:

CLAIMS:

2. Method according to claim 1, characterized in that the particles are metal-oxide-coated silicate or glass particles, which have been pretreated to achieve base solubility by extraction with mineral acids selected from the group consisting of, sulphuric acid, hydrochloric acid or nitric acid, together with an oxidizer, namely hydrogen peroxide.

5. Method according to claims 1, 2, 3 or 4, characterized in that the transparent metal oxide is titanium dioxide.

12. Pigment according to claim 9 or 10, characterized in that it contains at least 60% by weight of titanium dioxide.

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L11: Entry 3 of 27

File: USPT

Nov 20, 2001

US-PAT-NO: 6319513

DOCUMENT-IDENTIFIER: US 6319513 B1

TITLE: Oral liquid mucoadhesive compounds

DATE-ISSUED: November 20, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Dobrozsi, Douglas Joseph	Loveland	OH		

US-CL-CURRENT: 424/434; 424/435

CLAIMS:

What is claimed is:

1. A per oral or oral mucoretentive, aqueous liquid, pharmaceutical composition comprising:

(a) from about 2% to about 50%, by weight of the composition, of colloidal particles of titanium dioxide; and

(b) a safe and effective amount of a pharmaceutical active selected from the group consisting of gastrointestinal agents, analgesics, decongestants, expectorants, antitussives, antihistamines, bronchodilators, topical anesthetics, sensory agents, oral care agents, miscellaneous respiratory agents, and mixtures thereof;

wherein the composition has a sedimentation volume ratio of greater than about 0.90 when measured after about 48 hours, a triggered viscosity ratio of at least about 1.2; wherein the gastrointestinal agent is selected from the group consisting of anticholinergics, H₂-receptor antagonists, laxatives, gastroprotectants, gastrokinetic and prokinetic agents, proton pump inhibitors, antidiarrheals, agents effective for the treatment of H. pylori, polyanionic agents, plant extracts effective for the treatment of gastrointestinal disorders, and mixtures thereof.

2. The composition of claim 1 wherein the composition has a sedimentation volume ratio of greater than about 0.95, when measured after about 48 hours.

3. The composition of claim 2 wherein the composition has a sedimentation volume ratio of greater than about 0.98, when measured after about 48 hours.

4. The composition of claim 1 wherein the composition has a triggered viscosity ratio of at least about 1.4.

5. The composition of claim 4 wherein the composition has a triggered viscosity ratio of at least about 1.5.

6. The composition of claim 1 wherein the level of titanium dioxide is from about 3% to about 15%, by weight of the composition.

7. The composition of claim 6 wherein the titanium dioxide has a mean particle

size of less than about 1 micron.

8. The composition of claim 1 wherein the composition has a zero shear viscosity of greater than about 2,000 pascal seconds.

9. The composition of claim 8 wherein the composition has a zero shear viscosity of greater than about 7,500 pascal seconds.

10. An intranasal muco-retentive, aqueous liquid, pharmaceutical composition comprising:

(a) from about 2% to about 50%, by weight of the composition, of colloidal particles of titanium dioxide; and

(b) a safe and effective amount of a pharmaceutical active selected from the group consisting of gastrointestinal agents, analgesics, decongestants, expectorants, antitussives, antihistamines, bronchodilators, topical anesthetics, sensory agents, oral care agents, miscellaneous respiratory agents, and mixtures thereof;

wherein the composition has a sedimentation volume ratio of greater than about 0.90 when measured after about 48 hours and a triggered viscosity ratio of at least about 1.2.

11. The composition of claim 10 wherein the composition has a sedimentation volume ratio of greater than about 0.95, when measured after about 48 hours.

12. The composition of claim 11 wherein the composition has a sedimentation volume ratio of greater than about 0.98, when measured after about 48 hours.

13. The composition of claim 10 wherein the composition has a triggered viscosity ratio of at least about 1.4.

14. The composition of claim 13 wherein the composition has a triggered viscosity ratio of at least about 1.5.

15. The composition of claim 10 wherein the level of silica is from about 3% to about 15%, by weight of the composition.

16. The composition of claim 10 wherein the titanium dioxide has a mean particle size of less than about 1 micron.

17. The composition of claim 10 wherein the composition has a zero shear viscosity of greater than about 2,000 pascal seconds.

18. The composition of claim 17 wherein the composition has a zero shear viscosity of greater than about 7,500 pascal seconds.

19. A method of coating the alimentary canal by administering a safe and effective amount of the composition of claim 1.

20. A method of coating the nasal mucosa by administering a safe and effective amount of the composition of claim 10.

21. A method of preventing or treating symptoms of upper respiratory tract infections or upper respiratory tract tissue irritation or damage, by administering a safe and effective amount of the composition of claim 1.

22. A method of preventing or treating symptoms of upper respiratory tract infections or upper respiratory tract tissue irritation or damage, by administering a safe and effective amount of the composition of claim 10.

23. A method of administering an active agent to the alimentary canal by administering a safe and effective amount of the composition of claim 1.

24. A method of administering an active agent to the nasal mucosa by administering a safe and effective amount of the composition of claim 10.

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L11: Entry 3 of 27

File: USPT

Nov 20, 2001

DOCUMENT-IDENTIFIER: US 6319513 B1

TITLE: Oral liquid mucoadhesive compounds

Abstract Text (1):

The present invention relates to a per oral, oral, or intranasal pharmaceutical mucoretentive, aqueous liquid composition comprising from about 2% to about 50%, by weight of the composition, of colloidal particles of silica, titanium dioxide, clay, and mixtures thereof and a safe and effective amount of a pharmaceutical active selected from the group consisting of analgesics, decongestants, expectorants, antitussives, antihistamines, sensory agents, gastrointestinal agents, and mixtures thereof; wherein the composition has a sedimentation volume ratio of greater than about 0.90 and wherein the triggered viscosity ratio of the composition is at least about 1.2. The present invention further relates to a method of coating the alimentary canal or nasal mucosa, in particular to a method of preventing or treating symptoms of upper respiratory tract infections or upper respiratory tract tissue irritation or damage, by administering a safe and effective amount of the above composition.

Brief Summary Text (12):

(a) from about 2% to about 50%, by weight of the composition, of colloidal particles selected from the group consisting of silica, titanium dioxide, clay, and mixtures thereof; and

Detailed Description Text (7):

The present invention relates to mucoadhesive formulations comprising colloidal suspensions which form a coating matrix on the epithelium of the alimentary canal and/or the gastrointestinal tract. The term "colloidal" as used herein refers to finely divided solid material in which the particles of TiO_2 , SiO_2 , and/or clay (dispersed in another, liquid phase) have a particle size of generally less than 10 microns, or the particles have at least one dimension between about 1 and about 1000 nm. The particle size of the solid particles of the present invention are of colloidal dimension (about 1 nm to about 10 microns), preferably about 1,000 nm or smaller. The small particle size increases surface area for improved adsorption or bridging of the particle to mucin.

Detailed Description Text (25):

Preferably, the compositions of the present invention comprise only low levels of bioadhesive polymers, especially high molecular weight polymers, preferably less than about 1%, more preferably less than about 0.5%, even more preferably are essentially free of bioadhesive polymers, especially high molecular weight polymers; for example, those having a molecular weight of at least about 2,000 such as those disclosed in U.S. Pat. No. 5,458,879, Singh et al., issued Oct. 17, 1995, which is incorporated herein by reference in its entirety. Preferably, if the compositions of the present invention comprise a polymer, the ratio of colloidal particles (clay, silica, and/or titanium dioxide) to polymer is at least 10:1, preferably at least 20:1; more preferably, at least about 35:1 to 45:1.

Detailed Description Text (30):

The compositions of the present invention comprise a safe and effective amount of a particulate component which provides the mucoadhesive benefit. The particulate component comprises colloidal particles selected from the group consisting of silica, titanium dioxide, clay, and mixtures thereof.

Detailed Description Text (35):

The titanium dioxide is present at a level of from about 2% to about 50% by weight of the composition, preferably from about 3% to about 20%, more preferably from about 4% to about 9% by weight. Any of the available pharmaceutical grade forms of titanium dioxide are acceptable for use in the present invention as long as such form achieves the mucin interaction (T values) described above and efficiently achieves acceptable sedimentation volume ratio as specified herein. Such forms include rutile, anatase crystalline form, amorphous form, and any other form which is acceptable for the purposes of the present invention. These titanium dioxide particles can be chemically surface modified, for example with alumina, silica, or other stabilizing agent, to enhance the tissue barrier properties of the coating to hydrophilic substances.

Detailed Description Text (39):

The clay is present at a level of from about 2% to about 50 % by weight of the composition, preferably from about 3.5% to about 20%, more preferably from about 4.0% to about 10% by weight. Clays are composed of fine particles of clay minerals which are layer-type hydrous (containing structural hydroxyl groups) silicates of aluminum, magnesium, potassium, iron, and other less abundant elements, particularly alkalis and alkaline earth metals. Preferred are silicates of aluminum, magnesium and iron. More preferred are silicates of aluminum. Preferred is magnesium aluminum silicate (or aluminum magnesium silicate), occurring naturally in such smectite minerals as colerainite, saponite, and sapphirine. Refined magnesium aluminum silicates useful herein are readily available as Veegum, manufactured by R. T. Vanderbilt Company, Inc.

Detailed Description Text (40):

Clay may also contain varying amounts of non-clay minerals such as quartz, calcite, feldspar, and pyrite. Preferred clays useful herein are water swellable clays.

Detailed Description Text (41):

The term "clay" as used herein includes but is not limited to kaolin minerals such as kaolinite, china clay, dickite, nacrite, halloysite; serpentine minerals such as lizardite, halloysite, chrysotile, antigorite, carlosturanite, amestite, cronstedite, chamosite, berthierine, garierite; talc; pyrophyllite; ferripyrophyllite; smectites such as montmorillonites, beidellite, nontronite, hectorite, saponite, sauconite, medmontite, pimelite, bentonite; illite minerals such as ledikete, bravaisite, degraded mica, hydromica, hydromuscovite, hydrous illite, hydrous mica, K-mica, micaceous clay, and sericite; mica such as pegmatite, muscovite, and phlogopite; brittle mica such as margarite, and clintonite; glauconite; celadonite; chlorite and vermiculite such as pennine, clinochlore, chamosite, nimite, baileychlore, donbassite, cookite, sudoite, franklinfurnaceite; palygorskite and sepiolite minerals such as attapulgitite; allophane and imogolite; mixed layer clay minerals such as talc-chlorite; and mixtures thereof.

Detailed Description Text (42):

Preferred clays are selected from the group consisting of kaolin minerals, smectites, mica, and mixtures thereof. More preferred are clays selected from the group consisting of laponite, bentonite, hectorite, saponite, montmorillonites, and mixtures thereof.

Detailed Description Text (44):

Clays which are useful in the present invention include both mined, naturally occurring clays as well as synthetic clays. The clays must be pharmaceutically-acceptable. A more detailed description of the clays and clay minerals useful herein can be found in the following three references, each of which is incorporated by reference in its entirety: Kirk-Othmer, Encyclopedia of Chemical Technology, Fourth Edition, Vol. 6, pages 381-423; Dell, D. J., "Smectite Clays in Personal Care Products", Cosmetics & Toiletries, Vol. 108, May 1993, pages 79-85; and Theng B. K. G., "Formation and Properties of Clay-Polymer Complexes", Developments in Soil Science, Vol. 9. Clays include products available from Southern Clay Products, Gonzalez, Tex.; Generichem, Totowa, N.J.; R. T. Vanderbilt, Norwalk, Conn.; Smeotite, Inc., Casper, N.Y.

Detailed Description Text (107):

By "safe and effective amount" as used herein is meant an amount of silica, titanium dioxide, clay, or active agent, etc., high enough to significantly (positively) modify the condition to be treated or to effect the desired result, but low enough to avoid serious side effects (at a reasonable benefit/risk ratio), within the scope of sound medical/dental judgment. The safe and effective amount, will vary with the particular condition or disease being treated, the age and physical condition of the patient being treated, the severity of the condition, the duration of treatment, the nature of concurrent therapy, the specific form of the particles or active agent employed, and the particular vehicle from which the particles or active agent is applied.

Detailed Description Text (134):

Mix all of the water except 2% with the magnesium aluminum silicate and stir under high shear for at least 1 hour. Add glycerin and sorbitol and mix thoroughly. Add potassium bicarbonate and calcium carbonate with vigorous stirring until a uniform consistency is reached. Add sucrose and stir to obtain a uniform consistency. Dissolve the TK-10, monobasic potassium phosphate, vanilla cream flavor, and green color in the 2% remaining water. Add to the other ingredients and mix to obtain a uniform consistency. Combine the 0.16% benzyl alcohol with the parabens and add to the other ingredients and mix to obtain a uniform consistency. Combine mint flavor, 0.04% benzyl alcohol, and WS-3, and add to the other ingredients and mix to uniform consistency. Add hydrogen peroxide and mix approximately 10 minutes. The sedimentation volume ratio of this composition is 1. The zero shear viscosity is 256,800 pascal seconds. The triggered viscosity ratio is 2.37, and the high shear viscosity, at 100 per seconds, is 0.848 pascal seconds. The percent retained after 30 rinses in saliva by the method discussed above is 90.3%.

Detailed Description Text (159):

1.) Mix all of the water with the clay and stir under high shear for at least 1 hour to fully hydrate the clay.

Detailed Description Text (161):

3.) Add the cosolvent mixture with aromatic components to the hydrated clay and mix with gentle stirring at least 5 minutes. Add methyl salicylate, saccharin, and sodium lauryl sulfate and mix with low agitation for an additional 15 minutes.

Detailed Description Text (173):

Disperse the clay in one half of the total water by high shear mixing for at least 1 hour. Dissolve all other ingredients in chilled water by stirring. Filter this solution through a cellulose acetate membrane filter. Combine the filtered solution with the clay dispersion and stir for at least 10 minutes. Add flavor in an appropriate amount to provide a pleasant taste. Add camphor and eucalyptol in an appropriate amount to provide pleasant scent in use. Fill this final mixture into manually operated nasal spray pump bottles.

Detailed Description Paragraph Table (4):

COMPONENT % BY WT. Magnesium aluminum 7.5 silicate.sup.5 potassium bicarbonate 0.08 Calcium carbonate 7.72 glycerin 5.0 sorbitol 70% solution 11 sucrose 15 cooling agent TK-10 .RTM..sup.6 0.002 monobasic potassium 0.25 phosphate vanilla cream flavor 0.06 green color 0.001 benzyl alcohol 0.20 mint flavor 0.005 WS-3.sup.7 0.008 methyl paraben 0.05 propyl paraben 0.01 hydrogen peroxide 0.6 purified water q.s. to 100% .sup.5 Type IIA. .sup.6 Menthol, 3-1-menthoxy propane-1,2-diol manufactured by Takasago. .sup.7 N-ethyl-p-menthan-3-carboxamide.

CLAIMS:

1. A per oral or oral mucoretentive, aqueous liquid, pharmaceutical composition comprising:

(a) from about 2% to about 50%, by weight of the composition, of colloidal particles of titanium dioxide; and

(b) a safe and effective amount of a pharmaceutical active selected from the group consisting of gastrointestinal agents, analgesics, decongestants, expectorants, antitussives, antihistamines, bronchodilators, topical anesthetics, sensory agents,

oral care agents, miscellaneous respiratory agents, and mixtures thereof;

wherein the composition has a sedimentation volume ratio of greater than about 0.90 when measured after about 48 hours, a triggered viscosity ratio of at least about 1.2; wherein the gastrointestinal agent is selected from the group consisting of anticholinergics, H₂-receptor antagonists, laxatives, gastroprotectants, gastrokinetic and prokinetic agents, proton pump inhibitors, antidiarrheals, agents effective for the treatment of H. pylori, polyanionic agents, plant extracts effective for the treatment of gastrointestinal disorders, and mixtures thereof.

6. The composition of claim 1 wherein the level of titanium dioxide is from about 3% to about 15%, by weight of the composition.

7. The composition of claim 6 wherein the titanium dioxide has a mean particle size of less than about 1 micron.

10. An intranasal mucoretentive, aqueous liquid, pharmaceutical composition comprising:

(a) from about 2% to about 50%, by weight of the composition, of colloidal particles of titanium dioxide; and

(b) a safe and effective amount of a pharmaceutical active selected from the group consisting of gastrointestinal agents, analgesics, decongestants, expectorants, antitussives, antihistamines, bronchodilators, topical anesthetics, sensory agents, oral care agents, miscellaneous respiratory agents, and mixtures thereof;

wherein the composition has a sedimentation volume ratio of greater than about 0.90 when measured after about 48 hours and a triggered viscosity ratio of at least about 1.2.

16. The composition of claim 10 wherein the titanium dioxide has a mean particle size of less than about 1 micron.

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L11: Entry 3 of 27

File: USPT

Nov 20, 2001

US-PAT-NO: 6319513

DOCUMENT-IDENTIFIER: US 6319513 B1

TITLE: Oral liquid mucoadhesive compounds

DATE-ISSUED: November 20, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Dobrozsi, Douglas Joseph	Loveland	OH		

ASSIGNEE-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY	TYPE CODE
The Procter & Gamble Company	Cincinnati	OH			02

APPL-NO: 09/ 361533 [PALM]

DATE FILED: July 27, 1999

PARENT-CASE:

CROSS-REFERENCE TO RELATED APPLICATIONS This application claims the benefit of US Provisional Application No. 60/097,578, filed Aug. 24, 1998.

INT-CL: [07] A61 F 13/02

US-CL-ISSUED: 424/434; 424/435

US-CL-CURRENT: 424/434; 424/435

FIELD-OF-SEARCH: 424/434, 424/435

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

	PAT-NO	ISSUE-DATE	PATENTEE-NAME	US-CL
<input type="checkbox"/>	<u>3352752</u>	November 1967	Puetzer et al.	167/55
<input type="checkbox"/>	<u>4427681</u>	January 1984	Munshi	424/260
<input type="checkbox"/>	<u>5047244</u>	September 1991	Sanvordeker et al.	424/435
<input type="checkbox"/>	<u>5244667</u>	September 1993	Hagiwara et al.	424/409
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ART-UNIT: 215

PRIMARY-EXAMINER: Azpuru; Carlos

ABSTRACT:

The present invention relates to a per oral, oral, or intranasal pharmaceutical mucoretentive, aqueous liquid composition comprising from about 2% to about 50%, by weight of the composition, of colloidal particles of silica, titanium dioxide, clay, and mixtures thereof and a safe and effective amount of a pharmaceutical active selected from the group consisting of analgesics, decongestants, expectorants, antitussives, antihistamines, sensory agents, gastrointestinal agents, and mixtures thereof; wherein the composition has a sedimentation volume ratio of greater than about 0.90 and wherein the triggered viscosity ratio of the composition is at least about 1.2. The present invention further relates to a method of coating the alimentary canal or nasal mucosa, in particular to a method of preventing or treating symptoms of upper respiratory tract infections or upper respiratory tract tissue irritation or damage, by administering a safe and effective amount of the above composition.

24 Claims, 1 Drawing figures

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L11: Entry 9 of 27

File: USPT

Nov 18, 1997

US-PAT-NO: 5688492

DOCUMENT-IDENTIFIER: US 5688492 A

TITLE: Oral hygiene composition

DATE-ISSUED: November 18, 1997

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Galley; Edward	Nottingham			GB2
Cooper; Michael David	Nottingham			GB2

US-CL-CURRENT: 424/49; 424/421, 424/440, 424/464, 424/468, 424/48, 424/489, 424/52,
424/53, 424/54, 424/600, 424/604, 424/606, 424/617, 424/618, 424/630, 424/635,
424/641, 424/642, 424/643, 424/649, 424/650, 424/688, 424/691

CLAIMS:

We claim:

1. An oral composition suitable for use in oral hygiene or dental treatment comprising an effective amount of an antimicrobial agent in the form of a particulate, non-ion-exchanging, zeolite-free, inert metal oxide, a sparingly soluble metal salt providing antimicrobial metal ions in use and a further material selected from the group consisting of humectants, gelling agents, abrasives, fluoride sources, desensitizing agents, flavorings, colorings, sweeteners, preservatives, structuring agents, bactericides, anti-tartar agents and anti-plaque agents.
2. A composition as claimed in claim 1, wherein the metal salt is selected from the group consisting of silver, copper, gold, platinum and zinc salts.
3. A composition as claimed in claim 1, wherein the metal oxide is selected from the group consisting of titanium dioxide, aluminum oxide, zirconium oxide, hafnium oxide, niobium oxide, tantalum oxide, calcium hydroxyapatite, barium sulphate, and magnesium oxide.
4. A composition as claimed in claim 3, wherein the titanium dioxide is in at least one of anatase, rutile and brookite crystalline form.
5. A composition as claimed in claim 1 wherein the antimicrobial agent is employed in combination with a further antimicrobial agent providing short-term antimicrobial action.
6. A composition as claimed in claim 1 wherein the metal salt is present in an amount of from about 0.5 to 75% by weight of the antimicrobial agent.
7. A composition as claimed in claim 6 wherein the metal salt is present in an amount of from about 1 to 50% by weight of the antimicrobial agent.
8. A composition as claimed in claim 7 wherein the metal salt is present in an amount of from about 2 to 25% by weight of the antimicrobial agent.
9. A composition as claimed in claim 1 wherein the metal oxide has an average

particle size of less than about 25 micrometers.

10. A composition as claimed in claim 9 wherein the average particle size is between about 0.01 and 15 micrometers.

11. A composition as claimed in claim 1 wherein the metal oxide is titanium dioxide having a primary crystal size of from about 0.005 to 5 micrometers.

12. A composition as claimed in claim 11 wherein the titanium dioxide has a primary crystal size of from about 0.01 to 1 micrometers.

13. A composition as claimed in claim 1 wherein the metal oxide has a mean surface area of from about 1 to 300 m.² /g.

14. A composition as claimed in claim 1 wherein the antimicrobial agent is included in the composition in an amount of from about 1.times.10.⁻⁵ to 5% by weight.

15. A composition as claimed in claim 14 wherein the antimicrobial agent is included in the composition in an amount of from about 0.01% to 3% by weight.

16. A method of cleaning a mouth of an individual for cosmetic purposes, comprising applying to the mouth an oral hygiene composition comprising an effective amount of an antimicrobial agent comprising a particulate, non-ion-exchanging, zeolite-free, inert metal oxide and a sparingly soluble metal salt providing antimicrobial metal ions in use.

17. A method of treating or preventing dental caries or gingivitis, comprising applying to a mouth of an individual an oral hygiene composition comprising an effective amount of an antimicrobial agent comprising a particulate, non-ion-exchanging, zeolite-free, inert metal oxide and a sparingly soluble metal salt providing antimicrobial metal ions in use.

18. A method of providing sustained antimicrobial activity in a mouth of an individual, comprising applying to the mouth an oral hygiene composition comprising an effective amount of an antimicrobial agent comprising a particulate, non-ion-exchanging, zeolite-free, inert metal oxide and a sparingly soluble metal salt providing antimicrobial metal ions in use.

19. A composition as claimed in claim 5, wherein the further antimicrobial agent is selected from the group consisting of a zinc salt, a tin salt, cetyl pyridinium chloride, a bis-biguanide, aliphatic amines, bromochlorophene, hexachlorophene, salicylanilides, a quaternary ammonium compound and triclosan.

20. A composition as claimed in claim 1, wherein the composition is selected from the group consisting of a dentrifice, mouthwash, tooth powder, chewing gum, lozenge, mouth spray, denture cleansing formulation, tooth paint and glass ionomer cement.

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File: USPT

Nov 18, 1997

DOCUMENT-IDENTIFIER: US 5688492 A

TITLE: Oral hygiene composition

Brief Summary Text (12):

Preferably, the metal source is a metal compound, preferably a very sparingly soluble metal salt, suitably a silver salt such as silver chloride, since silver ions show particularly effective antimicrobial action. However, other antimicrobial metals such as copper, gold, platinum and zinc may also be used to good effect. The support material is preferably an inert oxide, preferably an inert metal oxide such as titanium dioxide (preferably in one or more of the crystalline forms anatase, rutile or brookite, suitably rutile) aluminium oxide, zirconium oxide or magnesium oxide.

Brief Summary Text (29):

Structuring agents may be required in, for example, dentifrices and gums to provide desirable textural properties and "mouthfeel". Suitable agents include natural gum binders such as gum tragacanth, xanthan gum, gum karaya and gum arabic, seaweed derivatives such as Irish moss and alginates, smectite clays such as bentonite or hectorite, carboxyvinyl polymers and water-soluble cellulose derivatives such as hydroxyethyl cellulose and sodium carboxymethyl cellulose. Improved texture may also be achieved, for example, by including colloidal magnesium aluminium silicate. Suitably, the structuring agent is included in an amount of from 0-5%, preferably 0-3% by weight of the oral hygiene composition.

Brief Summary Text (35):

Suitably, as described above, the compositions of the invention may include a further antimicrobial agent as a preservative and/or anti-plaque agent. Suitable antimicrobial agents include zinc salts (such as zinc citrate), cetyl pyridinium chloride, the bis-biguanides (such as chlorhexidine), aliphatic amines, bromochlorophene, hexachlorophene, salicylanilides, quaternary ammonium compounds and triclosan. Enzymic systems providing a source of a natural biocide may be used as alternatives to or in combination with the biocides listed. For example, a system comprising lactoperoxidase and glucose oxidase may be used to generate antimicrobial amounts of hydrogen peroxide in the presence of glucose, water and oxygen.

CLAIMS:

3. A composition as claimed in claim 1, wherein the metal oxide is selected from the group consisting of titanium dioxide, aluminum oxide, zirconium oxide, hafnium oxide, niobium oxide, tantalum oxide, calcium hydroxyapatite, barium sulphate, and magnesium oxide.

4. A composition as claimed in claim 3, wherein the titanium dioxide is in at least one of anatase, rutile and brookite crystalline form.

11. A composition as claimed in claim 1 wherein the metal oxide is titanium dioxide having a primary crystal size of from about 0.005 to 5 micrometers.

12. A composition as claimed in claim 11 wherein the titanium dioxide has a primary crystal size of from about 0.01 to 1 micrometers.

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L11: Entry 17 of 27

File: USPT

May 25, 1993

US-PAT-NO: 5214014

DOCUMENT-IDENTIFIER: US 5214014 A

TITLE: Deodorizing catalyst

DATE-ISSUED: May 25, 1993

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
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Nakatsuji; Tadao	Nara			JP
Nagano; Kazuhiko	Sakai			JP
Yoshida; Kimihiko	Sakai			JP

US-CL-CURRENT: 502/84; 502/182, 502/184, 502/185, 502/241, 502/324, 502/325,
502/330, 502/338, 502/345

CLAIMS:

What is claimed is:

1. A catalyst for use in a deodorizing method for removing malodorant components by oxidative destruction with ozone, the catalyst comprising a carrier and active components carried on said carrier in a thickness in the range of from 10 to 200 .mu.m, the active components for promoting the reaction of the malodorant components with said ozone comprising (i) about 20 to 90 per cent by weight of manganese dioxide; and (ii) clay.

2. A catalyst, for use in said deodorizing method, according to claim 1, wherein said catalyst comprises titanium dioxide in addition to said manganese dioxide and clay.

3. A catalyst, for use in said deodorizing method according to claim 1, wherein said catalyst comprises 1 to 30% of weight at least one metal oxide, the metal of which is selected from a group consisting of Cu, Co, Ni and Ag, in addition to said manganese dioxide and clay.

4. A catalyst for use in a deodorizing method for removing malodorant components by oxidative destruction with ozone, which comprises a carrier containing at least about 10% by weight of activated carbon, and active components disposed on said carrier in a thickness in the range of from 5 to 100 .mu.m, the active components comprising at least one metal, metal oxide or metal sulfate, the metal of which is selected from the group consisting of Ti, Cu, Mn, Ni, Fe, Ag, Au, Mo, Zr, Sn, Nb, and W.

5. A catalyst, for use in said deodorizing method, according to claim 4, wherein the thickness of said active components is 10 to 50 .mu.m.

6. A catalyst for use in a deodorizing method for removing malodorant components by oxidative destruction with ozone, the catalyst comprising a carrier and active components carried on said carrier in a thickness in the range of from 10 to 200 .mu.m, the active components for promoting the reaction of the malodorant components with said ozone comprising (i) 25 to 95 weight per cent, based on metal, of at least one metal oxide, the metal of which is selected from the

group consisting of Cu, Co, Mn, Ni and Fe; and (ii) 5 to 7 weight per cent, based on metal, of at least one member selected from the group consisting of titanium dioxide, silver oxide and gold, or an amount of silica which is sufficient to promote the destruction of malodorant components by reaction with ozone.

7. A catalyst for use in deodorizing method, according to claim 6, comprising as active components, at least one of MnO.sub.2 -TiO.sub.2, CuO-TiO.sub.2, Co.sub.3 O.sub.4 -TiO.sub.2, Fe.sub.2 O.sub.3 -TiO.sub.2, Fe.sub.2 O.sub.3 -Au, CuO-MnO.sub.2 -Ag.sub.2 O, MnO.sub.2 -Co.sub.3 O.sub.4 -TiO.sub.2, MnO.sub.2 -Co.sub.3 O.sub.4 -Ag.sub.2 O, NiO-MnO.sub.2 -TiO.sub.2, or MnO.sub.2 -SiO.sub.2.

8. A catalyst for use in the deodorizing method according to claim 6, wherein the concentration of said active components in said catalyst is at least 50 per cent by weight.

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L11: Entry 17 of 27

File: USPT

May 25, 1993

DOCUMENT-IDENTIFIER: US 5214014 A

TITLE: Deodorizing catalyst

Abstract Text (1):

The present invention provides a deodorizing method for removing malodorous components by oxidative destruction with ozone in the presence of a catalyst comprising a first component, which is a metal oxide, and a second component which is at least one member selected from the group consisting of titanium dioxide, silver oxide, silica and gold, or a catalyst comprising manganese dioxide and clay.

Brief Summary Text (8):

A solid type catalyst which may be wholly shaped of the catalytic active ingredient, e.g. manganese, titanium and clay in the form of a honeycomb, was used as an oxidative destruction catalyst to remove the malodorous components in an ozonolysis deodorizing method. Accordingly with conventional solid catalyst, however, since the saturated absorption of malodorous components in relation to the catalyst is large, when a condition change occurs, such as in a refrigeration or the like, for example, or when the reaction temperature rises, the concentration of malodorous components becomes low, the malodorous components are released from the catalyst to the air, and, as a result, concentration of malodorous components in the air is increased.

Brief Summary Text (13):

Examples of the active components include (i) at least one metal oxide, the metal of which is selected from the group consisting of copper, manganese, cobalt, iron and nickel, and (ii) at least one member selected from the group consisting of titanium dioxide, silver oxide, silica, and gold, or comprising manganese dioxide and clay.

Detailed Description Text (7):

In the catalyst comprising of manganese dioxide and clay, or manganese oxide, clay and titanium dioxide, a preferably concentration (% by weight) of the manganese dioxide is in the range from 20 to 90%. When concentration of the manganese dioxide is less than 20%, the catalyst does not have enough active component. When concentration of the manganese dioxide is more than 90%, capacity of the catalyst is not improved in proportion to addition of manganese dioxide.

Detailed Description Text (9):

The substituted catalyst mentioned above, includes MnO.sub.2 -CuO-clay, MnO.sub.2 -Co.sub.3 O.sub.4 -clay, MnO.sub.2 -Fe.sub.2 O.sub.3 -clay, MnO.sub.2 -NiO-clay, MnO.sub.2 -Ag2O-clay, MnO.sub.2 -CuO-clay-TiO.sub.2, MnO.sub.2 -Co.sub.3 O.sub.4 -clay-TiO.sub.2, MnO.sub.2 -Fe.sub.2 O.sub.3 -clay-TiO.sub.2, MnO.sub.2 -NiO-clay-TiO.sub.2, MnO.sub.2 -Ag2O-clay-TiO.sub.2 and the like. The above mentioned clay is suitably a bedded clay mineral which is mainly composed of pyrophyllite, talc, mica, chlorite, montmorillonite, kaolin, halloysite and the like. Examples of the clay are Kibushi clay, Gairome clay or the like.

Detailed Description Text (13):

Also, the catalyst of this invention can be produced by supporting active components on the carrier. The carrier is not particularly limited in shape, but may be in any form, such as honeycomb, pellet, cylinder, plate, pipe or the like. The carrier is not particularly limited in material, but may, for example, be made of urethane foam, clay or the like.

Detailed Description Text (22):

Examples of the catalytic active components having deodorization capability with ozone are at least one metal, at least one metal oxide, or at least one metal sulfate, the metal of which is selected from a group consisting of Ti, Cu, Mn, Ni, Fe, Ag, Au, Mo, Zr, Sn, Nb and W; and or the components may contain activated carbon or acid clay. The metal oxides to be carried includes those mainly comprising MnO.sub.2, NiO, CuO or Fe.sub.2 O.sub.3 ; those mainly comprising MnO.sub.2 -TiO.sub.2, MnO.sub.2 -CuO, MnO.sub.2 -Fe.sub.2 O.sub.3 ; those mainly comprising MnO.sub.2 -TiO.sub.2, MnO.sub.2 -CuO, MnO.sub.2 -Fe.sub.2 O.sub.3, MnO.sub.2 -Ag.sub.2 O, NiO-Co.sub.3 O.sub.4, NiO-TiO.sub.2, NiO-MnO.sub.2, NiO-Ag.sub.2 O, NiO-MoO.sub.3, NiO-WO.sub.3, NiO-SnO.sub.2; and those mainly comprising MnO.sub.2 -Ag.sub.2 O-TiO.sub.2, MnO.sub.2 -CuO-Ag₂O, NiO-MnO.sub.2 -Ag.sub.2 O, and NiO-MnO.sub.2 -TiO.sub.2, which have already been set forth by the inventors.

Detailed Description Text (28):

Kibusushi clay was dried for 18 hours at 100.degree. C., whereupon it was crushed in a sample mill having a 0.5 mm/.phi. screen. 20 kg of the resulting product was mixed with 1 kg of methyl cellulose type binder (Yuken Industry Co., YB-32) and water. The mixture was kneaded thoroughly by a neader. After that, the mixture was put into an orga-screw type extruder, to produce a honeycomb. In such a case, the water content was adjusted so as to have 30-35 kg/cm.sup.2 of pressure. The obtained honeycomb was air-dried at ambient temperature, and it was heated up to 500.degree. C. at a rate of 5.degree. C. per hour. After it was kept at 500.degree. C. for 3 hours, it was cooled down at a rate of 10.degree. C. per hour. Thus, a honeycomb type carrier having an open area of 64% and a pitch of 4.0 mm was obtained.

Detailed Description Text (68):

30 g of MnO.sub.2, having 48 m.sup.2 /g of specific surface area, and 70 g of anatase type TiO.sub.2, having 85 m.sup.2 /g of specific surface area, were added to 170 ml of a titania sol (TiO.sub.2 concentration: 150 g/l). The mixture was agitated and mixed for 30 minutes to produce a slurry, which was impregnated into a corrugated honeycomb made of ceramic fibers having 81% of open area and 4.0 mm of pitch. Thus, there was prepared a binary-catalyst carrying a layer of MnO.sub.2 -TiO.sub.2 (mol ratio of 24:76) of average thickness of about 50 .mu.m, with 101% of carry rate.

Detailed Description Text (86):

There was prepared a binary-catalyst carrying Fe.sub.2 O.sub.3 -Ag.sub.2 O (mol ratio of 95:5) having an average thickness of about 50 .mu.m with 104% of carry rate, in the same manner as Example 9, except that instead of 70 g of the anatase type TiO.sub.2 and 170 ml of a titania soluted used in Example 14, Au colloid, which was produced by adding 30% hydrogen peroxide to chloroauric acid at a pH of 8.0, was used in this Example 18.

Detailed Description Text (99):

704 g of MnO.sub.2, having 48 m.sup.2 /g of specific surface area, and 155 g of Kibushi clay were mixed with 1 l of water. To the mixture, 250 g of glass beads were added to adjust the degree of milling, and the mixture was agitated and mixed for 30 minutes to produce a slurry. After removing the glass beads the slurry was impregnated into a corrugated honeycomb made of ceramic fibers, having 81% open area and 4.0 mm of pitch. Thus, there was prepared a binary-catalyst carrying MnO.sub.2 -clay (weight ratio of 82:18) having an average thickness of about 50 .mu.m with 95% of carry rate.

Detailed Description Text (101):

There was prepared a ternary-catalyst carrying MnO.sub.2 -TiO.sub.2 -clay (weight ratio of 82:9:9) having an average thickness of about 50 .mu.m, with 95% of carry rate, in the same manner as Example 19, except that instead of 704 g of MnO.sub.2 and 155 g of Kibushi clay mixed with 1 l of water used in Example 19, 78 g of Kibushi clay and 517 ml of titania sol (TiO.sub.2 concentration : 150 g/l) was mixed with 500 ml of water in this Example 20.

Detailed Description Text (103):

There was prepared a binary-catalyst carrying MnO.sub.2 -clay (weight ratio of 24:76) having an average thickness of about 50 .mu.m with 103% of carry rate, in the same manner as Example 19, except that instead of 704 g of MnO.sub.2 and 155 g of

Kibushi clay mixed with 1 l of water used in Example 19, 95 g of Kibushi clay and 30 g of MnO.sub.2 were mixed with 500 ml of water in this Example 21.

Detailed Description Text (105):

There was prepared a ternary-catalyst carrying MnO.sub.2 -TiO.sub.2 -clay (weight ratio of 24:20:56) having an average thickness of about 50 .mu.m with 101% of carry rate, in the same manner as Example 21, except that instead of 95 g of Kibushi clay and 30 g of MnO.sub.2 mixed with 500 ml of water used in Example 21, 70 g of Kibushi clay, 30 g of MnO.sub.2 and 170 ml of titania sol were mixed with 500 ml of water in this Example 22.

Detailed Description Text (107):

There was prepared ternary-catalyst carrying MnO.sub.2 -CuO-clay (weight ratio of 77:5:18) having an average thickness of about 50 .mu.m with 97% of carry rate, in the same manner as Example 19, except that instead of 704 g of MnO.sub.2 used in Example 19, 43 g of CuO and 661 g of MnO.sub.2 were used in this Example 23.

Detailed Description Text (109):

There was prepared a ternary-catalyst carrying MnO.sub.2 -Co.sub.3 O.sub.4 -clay (weight ratio of 80:2:18) having an average thickness of about 50 .mu.m with 101% of carry rate, in the same manner as Example 19, except that instead of 704 g of MnO.sub.2 used in Example 19, 17 g of Co.sub.3 O.sub.4 and 687 g of MnO.sub.2 were used in this Example 24.

Detailed Description Text (111):

There was prepared a ternary-catalyst carrying MnO.sub.2 -Fe.sub.2 O.sub.3 -clay (weight ratio of 74:8:18) having an average thickness of about 50 .mu.m with 98% of carry rate, in the same manner as in Example 19, except that instead of 704 g of MnO.sub.2 used in Example 19, 70 g of Fe.sub.2 O.sub.3 and 634 g of MnO.sub.2 were used in this Example 25.

Detailed Description Text (113):

There was prepared a ternary-catalyst carrying MnO.sub.2 -NiO-clay (weight ratio of 76:6:18) having an average thickness of about 50 .mu.m with 100% of carry rate, in the same manner as Example 19, except that instead of 704 g of MnO.sub.2 used in Example 19, 50 g of NiO and 654 g of MnO.sub.2 were used in this Example 26.

Detailed Description Text (115):

There was prepared a ternary-catalyst carrying MnO.sub.2 -Ag.sub.2 O-clay (weight ratio of 80:2:18) having an average thickness of about 50 .mu.m with 100% of carry rate, in the same manner as Example 19, except that instead of 704 g of MnO.sub.2 used in Example 19, 17 g of Ag.sub.2 O and 687 g of MnO.sub.2 were used in this Example 27.

Detailed Description Text (117):

There was prepared a quaternary-catalyst carrying MnO.sub.2 -CuO-TiO.sub.2 -clay (weight ratio of 77:5:9:9) having an average thickness of about 50 .mu.m with 103% of carry rate, in the same manner as Example 23, except that instead of 155 g of Kibushi clay used in Example 19, 78 g of Gairome clay and 517 g of titania sol were used in this Example 28.

Detailed Description Text (119):

There was prepared a quaternary-catalyst carrying MnO.sub.2 -Ag.sub.2 O-TiO.sub.2 -clay (weight ratio of 80:2:9:9) having an average thickness of about 50 .mu.m with 103% of carry rate, in the same manner as Example 19, except that instead of 155 g of Kibushi clay used in Example 19, 78 g of Gairome clay and 517 g of titania sol were used in this Example 29.

Detailed Description Text (121):

100 g of Kibushi clay, having 56 m.sup.2 /g of specific surface area, was calcined for 3 hours at 500.C. Thus, a catalyst which consisted of Kibushi clay was obtained.

Detailed Description Text (128):

After Gairome clay was dried for 18 hours at 100.degree. C., it was crushed by a

sample mill having a screen which has holes of 0.55 mm diameter. Then, 8 kg of the clay thus crushed was mixed with 2 kg of activated carbon, 0.8 kg of methyl cellulose type binder (Yuken Industry Co., YB-32) and water. The mixture was kneaded thoroughly by a kneader and put into an orga-screw type extruder to obtain a honeycomb. After being air-dried at ambient temperatures, the honeycomb was heated up to 500.degree. C. at a rate of 5.degree. C./hour and kept in that temperature for 3 hours. Then the honeycomb was cooled down at a rate of 10.degree. C./hour. Thus, a honeycomb type carrier having 64% of open area and 1.0 mm of pitch was obtained.

Detailed Description Text (135):

A honeycomb shaped carrier was obtained in the same manner as Example 30, except that 5 kg of crushed Gairome clay, 5 kg of activated carbon, and 1 kg of methyl cellulose type binder (Yuken Industry, Co., YB-32) were used. The honeycomb was treated with the same slurry as in Example 31, and by adjusting the number of times of coating, 5 types of catalysts having thicknesses, respectively, of 5 .mu.m, 10 .mu.m, 18 .mu.m, 29 .mu.m and 43 .mu.m were obtained.

Detailed Description Text (143):

A catalyst carrying a layer of MnO.sub.2 having an average thickness of 57 .mu.m was obtained in the same manner as Example 33, except that 18 kg of Gairome clay and 2 kg of activated carbon were used in forming the carrier by extrusion.

Detailed Description Text (145):

A catalyst carrying a layer of MnO.sub.2 having an average thickness of 50 .mu.m, was obtained in the same manner as Example 30, except that 20 kg of Gairome clay was used, but activated carbon was not added in forming the carrier by extrusion.

Detailed Description Text (154):

30 g of MnO.sub.2, having a specific surface area of 48 m.sup.2 /g, and 70 g of anatase type TiO.sub.2, having a specific surface area of 85 m.sup.2 /g, were added to 170 ml of a titania sol (TiO.sub.2 concentration: 150 g/l). The mixture was agitated and mixed for 30 minutes to produce a slurry, which was impregnated into a corrugated honeycomb made of ceramic fibers having an open area of 81% and a pitch of 4.0 mm. Thus, there was prepared a binary-catalyst carrying about 50 .mu.m, in average thickness, of MnO.sub.2 -TiO.sub.2 (mol ratio of 24:76) with a carry rate of 101%.

Detailed Description Text (172):

There was prepared a binary-catalyst carrying about 50 .mu.m in average thickness of Fe.sup.2 O.sub.3 -Ag.sub.2 O (mol ratio of 95:5) with a carry rate of 104%, in the same manner of Example 38, except that a Au colloid, which was produced by adding 30% hydrogen peroxide to chloroauric acid at a pH of 8.0, was used in Example 16, instead of 70 g of anatase type TiO.sub.2 and 170 ml of a titania solution used in Example 43.

Detailed Description Text (185):

704 g of MnO.sub.2, having a specific surface area of 48 m.sup.2 /g, and 155 g of Kibushi clay were mixed with 1 l of water. To the mixture, 250 g of glass beads were added to adjust the degree of milling, and was agitated and mixed for 30 minutes to produce a slurry. After removing the glass beads, the slurry was impregnated into a corrugated honeycomb made of ceramic fibers having an open area rate of 81% and pitch of 4.0 mm. Thus, there was prepared a binary-catalyst carrying about 50 .mu.m in average thickness of MnO.sub.2 -clay (weight rate of 82:18) with a carry rate of 95%.

Detailed Description Text (187):

There was prepared a ternary-catalyst carrying about 50 .mu.m in average thickness of MnO.sub.2 -TiO.sub.2 -clay (weight rate of 82:9:9) with a carry rate of 95%, in the same manner as that of Example 48, except that 78 g of Kibushi clay and 517 ml of titania sol (TiO.sub.2 concentration: 150 g/l) was mixed with 500 ml of water in this Example 49, instead of 704 g of MnO.sub.2 and 155 g Kibushi clay mixed with 1 l of water in Example 17.

Detailed Description Text (189):

There was prepared a binary-catalyst carrying 50 .mu.m in average thickness of

MnO.sub.2 -clay (weight ratio of 24:76) with a carry rate of 103%, in the same manner as that set forth in Example 48, except that 95 g of Kibushi clay and 30 g of MnO.sub.2 were mixed with 500 ml of water in this Example 50, instead of the 704 g of MnO.sub.2 and 155 g Kibushi clay mixed with 1 l of water in Example 48.

Detailed Description Text (191):

There was prepared a ternary-catalyst carrying 50 .mu.m in average thickness of MnO.sub.2 -TiO.sub.2 -clay (weight ratio of 24:20:56) with a carry rate of 101%, in the same manner as the Example 50, except that 70 g of Kibushi clay, 30 g of MnO.sub.2 and 170 ml of titania sol was mixed with 500 ml of water in this Example 51, instead of 95 g of Kibushi clay and 30 g of MnO.sub.2 mixed with 500 ml of water in Example 50.

Detailed Description Text (193):

There was prepared a ternary-catalyst carrying 50 .mu.m in average thickness of MnO.sub.2 -CuO-clay (weight ratio of 77:5:18) with a carry rate of 97%, in the same manner as that Example 48, except that 43 g of CuO and 661 g of MnO.sub.2 used in this Example 52, instead of 704 g of MnO.sub.2 used in Example 48.

Detailed Description Text (195):

There was prepared a ternary-catalyst carrying 50 .mu.m in average thickness of MnO.sub.2 -Co3O4-clay (weight ratio of 80:2:18) with a carry rate of 101%, in the same manner as that Example 48, except that 17 g of Co.sub.3 O.sub.4 and 687 g of MnO.sub.2 used in this Example 53, instead of 704 g of MnO.sub.2 used in Example 48.

Detailed Description Text (197):

There was prepared a ternary-catalyst carrying 50 .mu.m in average thickness of MnO.sub.2 -Fe.sub.2 O.sub.3 -clay (weight ratio of 74:8:18) with a carry rate of 98%, in the same manner as that Example 48, except that 70 g of Fe.sub.2 O.sub.3 and 634 g of MnO.sub.2 used in this Example 54, instead of 704 g of MnO.sub.2 used in Example 48.

Detailed Description Text (199):

There was prepared a ternary-catalyst carrying 50 .mu.m in average thickness of MnO.sub.2 -NiO-clay (weight ratio of 76:6:18) with a carry rate of 100%, in the same manner as that Example 48, except that 50 g of NiO and 654 g of MnO.sub.2 used in this Example 55, instead of 704 g of MnO.sub.2 used in Example 48.

Detailed Description Text (201):

There was prepared a ternary-catalyst carrying 50 .mu.m in average thickness of MnO.sub.2 -Ag.sub.2 O-clay (weight ratio of 80:2:18) with a carry rate of 100%, in the same manner as that Example 48, except that 17 g of Ag.sub.2 O and 687 g of MnO.sub.2 used in this Example 56, instead of 704 g of MnO.sub.2 used in Example 48.

Detailed Description Text (203):

There was prepared a quaternary-catalyst carrying 50 .mu.m in average thickness of MnO.sub.2 -CuO-TiO.sub.2 -clay (weight ratio of 77:5:9:9) with a carry rate of 103%, in the same manner as that Example 52, except that 78 g of Gairome clay and 517 g of titania sol used in this Example 57, instead of 155 g of Kibushi clay used in Example 48.

Detailed Description Text (205):

There was prepare a quaternary-catalyst carrying 50 .mu.m in average thickness of MnO.sub.2 -Ag.sub.2 O-TiO.sub.2 -clay (weight ratio of 80:2:9:9) with a carry rate of 103%, in the same manner as that Example 48, except that 78 g of Gairome clay and 517 g of titania sol used in this Example 58, instead of 155 g of Kibushi clay in Example 48.

Detailed Description Text (207):

100 g of Kibushi clay having a specific surface area of 56 m.sup.2 /g was calcined for 3 hours at 500.degree. C. Thus, a catalyst consisting of Kibushi clay, was obtained.

2. A catalyst, for use in said deodorizing method, according to claim 1, wherein said catalyst comprises titanium dioxide in addition to said manganese dioxide and clay.

3. A catalyst, for use in said deodorizing method according to claim 1, wherein said catalyst comprises 1 to 30% of weight at least one metal oxide, the metal of which is selected from a group consisting of Cu, Co, Ni and Ag, in addition to said manganese dioxide and clay.

6. A catalyst for use in a deodorizing method for removing malodorous components by oxidative destruction with ozone, the catalyst comprising a carrier and active components carried on said carrier in a thickness in the range of from 10 to 200 .mu.m, the active components for promoting the reaction of the malodorous components with said ozone comprising (i) 25 to 95 weight per cent, based on metal, of at least one metal oxide, the metal of which is selected from the group consisting of Cu, Co, Mn, Ni and Fe; and (ii) 5 to 7 weight per cent, based on metal, of at least one member selected from the group consisting of titanium dioxide, silver oxide and gold, or an amount of silica which is sufficient to promote the destruction of malodorous components by reaction with ozone.

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L12: Entry 2 of 33

File: USPT

Sep 10, 2002

DOCUMENT-IDENTIFIER: US 6447563 B1

TITLE: Chemical mechanical polishing slurry system having an activator solution

Brief Summary Text (7):

In the case of CMP of metals, the chemical action is generally considered to take one of two forms. In the first mechanism, the chemicals in the solution react with the metal layer to continuously form an oxide layer on the surface of the metal. This generally requires the addition of an oxidizer to the solution such as hydrogen peroxide, ferric nitrate, etc. Then the mechanical abrasive action of the particles continuously and simultaneously removes this oxide layer. A judicious balance of these two processes obtains optimum results in terms of removal rate and polished surface quality.

Brief Summary Text (10):

CMP metal slurries, on the other hand, are two part mixtures consisting of a dispersion and an oxidizer. The dispersion comprises an abrasive, an acid to lower the pH to about 2 to 6, optionally a surfactant which maintains the abrasive in suspension and other chemicals tailored to the metal being polished. An example is a tungsten layer slurry called Biplanar.RTM. made by EKC. The dispersion is an acidic dispersion (approximately pH of 3, with 5 to 15% alumina particles). Acids reportedly used in the slurry include carboxylic acids or nitric acid. At the point of use, the dispersion is mixed with an oxidizer, such as hydrogen peroxide or ferric nitrate, to form the slurry that will be used to polish the metal layers.

Brief Summary Text (27):

The novel activator comprises chemicals that are customized to the particular metal being polished. For example, the activator of the present invention may include an oxidizer. Any suitable oxidizer may be used. Examples of a suitable oxidizer are hydrogen peroxide, potassium ferricyanide, potassium dichromate, potassium iodate, potassium bromate, vanadium trioxide, hypochlorous acid, sodium hypochlorite, potassium hypochlorite, calcium hypochlorite, magnesium hypochlorite, ferric nitrate, KMgO.sub.4, and mixtures thereof. The preferred oxidizer is hydrogen peroxide. The amount of oxidizer is typically 0.01% to 10%, and preferably 0.1 to 5% by weight of the total weight of the slurry.

Brief Summary Text (35):

In a preferred embodiment, the activator solution comprises: (a) about 0.05 wt. % to 5 wt. % of hydrogen peroxide; (b) about 0.05 wt. % to 3 wt. % of propanoic acid; (c) about 0.02 wt. % to 1.5 wt. % of benzotriazole; and (d) about 0.01 wt. % to 2 wt. % of ethanolamine.

Detailed Description Text (4):

A copper slurry was prepared by utilizing a silica dispersion containing 12% fumed silica. The silica dispersion was mixed with an activator containing hydrogen peroxide and propanoic acid. The final copper slurry mixture contained 1% H.sub.2O.sub.2 by weight, 4% fumed silica by weight and 0.1 molar propanoic acid. Copper wafers were obtained by sputter deposition on a silicon wafer and were polished using the copper slurry with a Rodel IC1400 pad and IPEC 472 tool. The removal rates were in excess of 450 nm, the non-uniformity was less than 5% and the selectivity of copper to SiO2 was over 200. The passive etch rate without mechanical polishing was 10 nm/minute. In comparison, a commercial copper slurry (Rodel QC 1020) had similar performance.

CLAIMS:

2. The system of claim 1 wherein said abrasive is selected from the group consisting of: silica, alumina, silicon carbide, silicon nitride, iron oxide, ceria, zirconium oxide, tin oxide, titanium dioxide and mixtures thereof.

5. The system of claim 1 wherein said acid is selected from the group consisting of: formic acid, acetic acid, propanoic acid, butanoic acid, pentanoic acid, hexanoic acid, heptanoic acid, octanoic acid, nonanoic acid, lactic acid, hydrochloric acid, nitric acid, phosphoric acid, sulfuric acid, hydrofluoric acid, malic acid, tartaric acid, gluconic acid, citric acid, phthalic acid, pyrocatechoic acid, pyrogallol carboxylic acid, gallic acid, tannic acid and mixtures thereof.

15. The activator of claim 14 wherein said acid is selected from the group consisting of: formic acid, acetic acid, propanoic acid, butanoic acid, pentanoic acid, hexanoic acid, heptanoic acid, octanoic acid, nonanoic acid, lactic acid, hydrochloric acid, nitric acid, phosphoric acid, sulfuric acid, hydrofluoric acid, malic acid, tartaric acid, gluconic acid, citric acid, phthalic acid, pyrocatechoic acid, pyrogallol carboxylic acid, gallic acid, tannic acid and mixtures thereof.

20. The activator of claim 14 wherein said metal layer is tungsten, said oxidizer is selected from the group consisting of: hydrogen peroxide and ferric nitrate, and said acid is selected from the group consisting of: lactic acid and nitric acid.

22. The activator of claim 14 wherein said metal layer is copper and wherein said activator comprises hydrogen peroxide; an acid selected from the group consisting of propanoic acid, phthalic acid and citric acid; and benzotriazole.

23. The activator of claim 14 wherein said metal layer is tantalum and wherein said activator comprises hydrogen peroxide, ammonium fluoride and at least one other component.

24. The activator of claim 14 wherein said metal layer is aluminum and wherein said activator comprises hydrogen peroxide, ammonium fluoride and a weak acid.

25. The activator of claim 14 wherein said metal layer is titanium and wherein said activator comprises hydrogen peroxide, ammonium fluoride and a weak acid.

26. An activator solution, free of abrasives, for a slurry system used for polishing metal layers, said slurry system having a first part and a second part, said activator solution comprising: (a) about 0.05 wt. % to 5 wt. % of hydrogen peroxide; (b) about 0.05 wt. % to 3 wt. % of propanoic acid; (c) about 0.02 wt. % to 1.5 wt. % of benzotriazole; and (d) about 0.01 wt. % to 2 wt. % of ethanolamine; wherein said first part is a dispersion solution and said activator solution is said second part of said slurry system, and said activator solution is packaged separately from said dispersion solution.

28. The process of claim 27 wherein said abrasive is selected from the group consisting of: silica, alumina, silicon carbide, silicon nitride, iron oxide, ceria, zirconium oxide, tin oxide, titanium dioxide and mixtures thereof.

31. The process of claim 27 wherein said acid is selected from the group consisting of: formic acid, acetic acid, propanoic acid, butanoic acid, pentanoic acid, hexanoic acid, heptanoic acid, octanoic acid, nonanoic acid, lactic acid, hydrochloric acid, nitric acid, phosphoric acid, sulfuric acid, hydrofluoric acid, malic acid, tartaric acid, gluconic acid, citric acid, phthalic acid, pyrocatechoic acid, pyrogallol carboxylic acid, gallic acid, tannic acid and mixtures thereof.

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L12: Entry 3 of 33

File: USPT

Jul 16, 2002

DOCUMENT-IDENTIFIER: US 6420437 B1

TITLE: Titanium oxide colloidal sol and process for the preparation thereof

Brief Summary Text (11):

As a means for solving the above-mentioned problems, Japanese Unexamined Patent Publication No. 9-71,418 discloses a sol liquid comprising hydrogen peroxide and titanium dioxide and a method of producing the sol liquid. The sol liquid is advantageous in that the sol liquid can be neutralized. However, since an oxidizing agent is contained, the sol liquid is disadvantageous in that the effect of the sol liquid for preventing the metal corrosion is low, the sol liquid is colored yellow, and a colorless coating is difficult to be formed from the sol liquid unless the coating is heat-dried.

CLAIMS:

1. A titanium dioxide colloid sol comprising 50 to 100 parts by weight of titanium dioxide colloidal particles charged with negative electricity and 5 to 50 parts by weight of a complexing agent and 1 to 50 parts by weight of an alkaline substance, the complexing agent comprising at least one member selected from the group consisting of condensed phosphoric acids and condensed phosphate salts.
2. The titanium dioxide colloid sol as claimed in claim 1, wherein the pH value of the titanium dioxide sol is 5 to 10.
3. The titanium dioxide colloid sol as claimed in claim 1, wherein the alkaline substance comprises at least one member selected from the group consisting of ammonium compounds, alkali metal compounds and amine compounds.
4. The titanium dioxide colloid sol as claimed in claim 1, wherein the alkaline substance comprises at least one member selected from the group consisting of oxazine, piperidine and choline compounds.
5. A method of producing a titanium dioxide colloid sol characterized by mixing an alkaline substance into an acid titanium dioxide colloid sol containing 50 to 100 parts by weight of titanium dioxide colloid sol and 5 to 50 parts by weight of a complexing agent, to adjust the pH value of the sol to a level of 5 to 10, and to thereby cause the titanium dioxide colloidal particles to be charged with negative electricity, the complexing agent comprising at least one member selected from the group consisting of condensed phosphoric acid and condensed phosphate salts.
6. The method of producing the titanium dioxide colloid sol as claimed in claim 5, wherein the alkaline substance comprises at least one member selected from the group consisting of ammonium compounds, alkali metal compounds and amine compounds.
7. The method of producing the titanium dioxide colloid sol as claimed in claim 5, wherein the alkaline substance comprises at least one member selected from the group consisting of oxazine, piperidine and choline compounds.
8. A method of producing a titanium dioxide colloid sol, characterized by mixing an alkaline substance into an acid titanium dioxide colloid sol comprising 50 to 100 parts by weight of titanium dioxide colloidal particles and 5 to 50 parts by weight of a complexing agent to adjust the pH value of the resultant sol to a level of 6 to 12, and applying a deionization treatment to the sol to thereby cause the titanium

dioxide colloidal particles to be charged with negative electricity.

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L12: Entry 22 of 33

File: USPT

Jan 28, 1997

DOCUMENT-IDENTIFIER: US 5597554 A

TITLE: Oral hygiene system

Brief Summary Text (22):

The peroxide compound containing dentifrice may comprise a tooth whitening dentifrice as disclosed in U.S. Pat. No. 5,084,268, such as the commercially available hydrogen peroxide dentifrice sold under the registered trademark STAY WHITE.RTM.. STAY WHITE.RTM. dentifrice is a non-abrasive dentifrice comprising a blend of from about 2% to 35% corn starch, which functions as a jelling agent, a thickener, a filler and a binder, from 5% to 30% sorbitol which functions as a humectant and sweetening agent, from 0.2% to 10% hydrogen peroxide, from 0.2% to 4% Carbomer 940 which comprises a gum and jelling agent, 0.1% to 2% flavor, from 0.2% to 2% sodium laurel sulfate, which functions as a foaming and surfactant agent, from 0% to 0.25% potassium sorbate which functions as a preservative, 0.1% to 0.25% sodium benzoate which functions as a preservative and deionized water in a sufficient quantity. Additionally, a water softening chelating agent such as EDTA (approximately 0.1%) may be employed.

Brief Summary Text (24):

U.S. Pat. No. 4,980,152 discloses a non-abrasive aqueous oral gel composition comprising from about 0.5 to about 10% by weight urea peroxide or hydrogen peroxide and from about 0.01 to 2% by weight of a fluoride providing compound. The composition further includes a thickening agent such as carboxy polymethylene, a non-ionic surfactant such as PLURONIC F127, alkali soluble cellulose ethers as viscosity increasing agents, potassium phosphate as a buffering agent and glycerine as a carrier and flavoring and sweetening agents.

Brief Summary Text (25):

U.S. Pat. No. 4,839,156 discloses an aqueous dental gel containing 18-25% by weight of a polyoxyethylene polypropylene block copolymer gelling agent, hydrogen peroxide, 15-40% by weight of a polyethylene glycol humectant, flavor, sweetening agent and a non-ionic surfactant as the essential ingredients.

Brief Summary Text (26):

U.S. Pat. Nos. 4,522,805 and 4,567,036 disclose a toothpaste containing urea peroxide which dissociates into urea and hydrogen peroxide in the oral cavity, in a paste carrier comprising an anionic detergent, sorbitol and glycerin humectant and a thickening agent such as gum tragacanth, sodium alginate or sodium carboxymethyl cellulose.

Detailed Description Text (17):

As with Example 1, the abrasive constituent in the above toothpaste formulation is Hydrated Silica or Aluminum Hydroxide. The Hydrated Silica or Aluminum Hydroxide contained therein is in a higher concentration than in Example 1. The toothpaste formulation of Example 2 utilizes a higher percentage of abrasive material, Hydrated Silica for the purpose of providing a tartar control or anti-tartar abrasive formulation. Additional constituents of such formulation which may provide anti-tartar characteristics include Tetrasodium Pyrophosphate and PVM/MA Copolymer.

Detailed Description Text (29):

It should be appreciated that the system of the present invention functions with any extrudable dentifrice formulation having a peroxide compound constituent. Included among such dentifrices are those containing a hydrogen peroxide constituent such as

the dentifrices disclosed in U.S. Pat. No. 5,084,268, incorporated herein by reference; U.S. Pat. No. 4,980,152; U.S. Pat. No. 4,839,156 and Examples A, B, C, and E. Also included are dentifrices containing urea peroxide, such as Examples D and F, dentifrices containing carbamide peroxide, such as Example G and dentifrices containing calcium peroxide, such as those disclosed in U.S. Pat. No. 4,405,599 and U.S. Pat. No. 4,980,154, both of which are incorporated herein by reference. Numerous additional peroxide compound dentifrice formulations are commercially available.

Detailed Description Text (30):

Pursuant to the invention, equal volumes of peroxide containing dentifrice and conventional toothpaste are extruded onto the ends of bristles of a toothbrush and the toothbrush is inserted into the oral cavity with the user brushing teeth interdental spaces and gingival tissue in a normal fashion with conventional brush strokes. It has been found that the constituents of conventional toothpaste present an environment which accelerates the breakdown of the peroxide compound constituent and the generation of active oxygen within the oral cavity. At least one factor involved in the mechanism involving the accelerated breakdown of the peroxide constituent of the dentifrice is the fact that conventional toothpastes include pH elevating constituents and at elevated pH levels, hydrogen peroxide decomposes more readily.

Detailed Description Text (31):

It is believed that a major factor in the accelerated breakdown of the hydrogen peroxide constituent is the abrasive constituent of the conventional toothpaste. In many instances, the abrasive constituent of a conventional toothpaste comprises Hydrated Silica (Example 1, Example 2, Example 3 and Example 5). In other instances, the abrasive constituent comprises Calcium Carbonate (Example 4 and Example 7). While in other toothpaste formulations, the abrasive constituent comprises Dicalcium Phosphate Dihydrate (Example 6). These abrasive compounds comprise metal salts which reduce the peroxide to initiate the release of oxygen. Further conventional toothpaste constituents which are believed to accelerate the decomposition of Hydrogen Peroxide include Sodium Fluoride, Sodium Monofluorophosphate, Tetrasodium Pyrophosphate, Tetrasodium Phosphate, Titanium Dioxide, Cellulose Gum, Sodium Hydroxide, Trisodium Phosphate and Sodium Phosphate.

Detailed Description Text (32):

It has also been observed that brushing with a mixture of both conventional toothpaste and a dentifrice having a Hydrogen Peroxide constituent results in enhancement of the flavoring of the conventional toothpaste. While the exact mechanism of such enhancement is unknown, it is believed that the generation of free oxygen within the oral cavity includes, among its beneficial effects, the enhancement of taste sensitivity.

Detailed Description Paragraph Table (1):

	Ingredient	Wt. %
	PLURONIC F127	20.000
	Glycerin	40.000
	<u>Hydrogen Peroxide</u> (35%)	4.285
	Methyl Salicylate	0.500
	FD&C Blue	0.005
	<u>Phosphoric Acid</u> (85% w/w)	0.150
	Deionized water	q.s.

Detailed Description Paragraph Table (2):

	Ingredient	Parts
	<u>Hydrogen peroxide</u> (35%)	14.30
	Purified water	84.45
	CARBOPOL 934	0.50
	Hydroxyethyl cellulose	0.50
	Triethanolamine	0.25

Detailed Description Paragraph Table (3):

	Ingredient	Parts
	<u>Hydrogen peroxide</u> (35%)	11.5
	Distilled water	86.6
	CARBOPOL 934	1.5
	Sodium lauryl sulfate	0.1
	Hydroxypropyl cellulose	0.3
	Sodium hydroxide (10%)	q.s. (pH 3.0-4.5)

Detailed Description Paragraph Table (5):

	Ingredients	Parts
	<u>Hydrogen Peroxide</u> (35%)	10.00
	CARBOPOL 940	

1.50 Polyethylene Glycol 400 30.00 Glycerine 99.6% 25.00 Butylated Hydroxy Anisole
 0.50 Sodium Saccharin 0.50 Mixed Flavor oils 1.00 Calcium Pyrophosphate 29.00 Sodium
 Lauryl Sarcosinate 1.50 Tetrasodium Pyrophosphate 1.00

Detailed Description Paragraph Table (9):

<u>Ingredient Parts</u>	
Hydroxide 40.0	Glycerin 25.0 Sorbitol 15.0 <u>Tetrasodium Pyrophosphate</u> 5.0 Sodium
Lauryl Sulfate 1.5	Cellulose Gum 1.2 PEG-12 1.0 PVM/MA Copolymer 1.0 Sodium Fluoride
0.2 Sodium Saccharin 0.2	Sodium Hydroxide 0.1 Flavor 0.7 Colorant q.s.

Detailed Description Paragraph Table (13):

<u>Ingredient Parts</u>	
Glycerin 35.0	Sodium Lauryl Sulfate 1.5 Cellulose Gum 1.2 Sodium Monofluorophosphate
0.7 Flavor 0.75	Sodium Benzoate 0.2 <u>Tetrasodium Pyrophosphate</u> 0.2 Sodium Saccharin
0.2	

CLAIMS:

4. The method of claim i wherein the conventional toothpaste also contains one or more compounds selected from the group consisting of sodium fluoride, sodium monofluorophosphate, tetrasodium pyrophosphate, tetrasodium phosphate, titanium dioxide, cellulose gum, sodium hydroxide, trisodium phosphate, and sodium phosphate, in an amount sufficient to accelerate decomposition of the peroxide compound.
6. The method of claim 1 wherein the peroxide compound is selected from the group consisting of hydrogen peroxide, urea peroxide, carbamide peroxide, and calcium peroxide.
10. The method of claim 7 wherein the conventional toothpaste also contains one or more compounds selected from the group consisting of sodium fluoride, sodium monofluorophosphate, tetrasodium pyrophosphate, tetrasodium phosphate, titanium dioxide, cellulose gum, sodium hydroxide, trisodium phosphate, and sodium phosphate, in an amount sufficient to accelerate decomposition of the peroxide compound.
12. The method of claim 7 wherein the peroxide compound is selected from the group consisting of hydrogen peroxide, urea peroxide, carbamide peroxide, and calcium peroxide.

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L4: Entry 4 of 37

File: USPT

Jul 16, 2002

US-PAT-NO: 6420437

DOCUMENT-IDENTIFIER: US 6420437 B1

TITLE: Titanium oxide colloidal sol and process for the preparation thereof

DATE-ISSUED: July 16, 2002

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
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Nakamura; Mitsuru	Tokyo			JP
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US-CL-CURRENT: 516/90; 106/13, 106/287.19, 106/440, 106/448, 252/588, 502/350

CLAIMS:

What the claimed is:

1. A titanium dioxide colloid sol comprising 50 to 100 parts by weight of titanium dioxide colloidal particles charged with negative electricity and 5 to 50 parts by weight of a complexing agent and 1 to 50 parts by weight of an alkaline substance, the complexing agent comprising at least one member selected from the group consisting of condensed phosphoric acids and condensed phosphate salts.
2. The titanium dioxide colloid sol as claimed in claim 1, wherein the pH value of the titanium dioxide sol is 5 to 10.
3. The titanium dioxide colloid sol as claimed in claim 1, wherein the alkaline substance comprises at least one member selected from the group consisting of ammonium compounds, alkali metal compounds and amine compounds.
4. The titanium dioxide colloid sol as claimed in claim 1, wherein the alkaline substance comprises at least one member selected from the group consisting of oxazine, piperidine and choline compounds.
5. A method of producing a titanium dioxide colloid sol characterized by mixing an alkaline substance into an acid titanium dioxide colloid sol containing 50 to 100 parts by weight of titanium dioxide colloid sol and 5 to 50 parts by weight of a complexing agent, to adjust the pH value of the sol to a level of 5 to 10, and to thereby cause the titanium dioxide colloidal particles to be charged with negative electricity, the complexing agent comprising at least one member selected from the group consisting of condensed phosphoric acid and condensed phosphate salts.
6. The method of producing the titanium dioxide colloid sol as claimed in claim 5, wherein the alkaline substance comprises at least one member selected from the group consisting of ammonium compounds, alkali metal compounds and amine compounds.
7. The method of producing the titanium dioxide colloid sol as claimed in claim 5, wherein the alkaline substance comprises at least one member selected from the group consisting of oxazine, piperidine and choline compounds.

8. A method of producing al titanium dioxide colloid sol, characterized by mixing an alkaline substance into an acid titanium dioxide colloid sol comprising 50 to 100 parts by weight of titanium dioxide colloidal particles and 5 to 50 parts by weight of a complexing agent to adjust the pH value of the resultant sol to a level of 6 to 12, and applying a deionization treatment to the sol to thereby cause the titanium dioxide colloidal particles to be charged with negative electricity.

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L4: Entry 4 of 37

File: USPT

Jul 16, 2002

DOCUMENT-IDENTIFIER: US 6420437 B1

TITLE: Titanium oxide colloidal sol and process for the preparation thereof

Brief Summary Text (11):

As a means for solving the above-mentioned problems, Japanese Unexamined Patent Publication No. 9-71,418 discloses a sol liquid comprising hydrogen peroxide and titanium dioxide and a method of producing the sol liquid. The sol liquid is advantageous in that the sol liquid can be neutralized. However, since an oxidizing agent is contained, the sol liquid is disadvantageous in that the effect of the sol liquid for preventing the metal corrosion is low, the sol liquid is colored yellow, and a colorless coating is difficult to be formed from the sol liquid unless the coating is heat-dried.

Detailed Description Text (31):

As explained in detail above, the titanium dioxide colloid sol of the present invention is a neutral photocatalytic titanium dioxide colloid sol having high stability and dispersibility and usable as a coating material for articles, for example, metal articles having a low resistance to corrosion and organic articles having a low resistance to deterioration. When the titanium dioxide colloid sol is employed for the purpose of decomposition of stains, ultraviolet ray absorption, sterilization, gas decomposition, and water treatment, the sol can be applied to wide various materials, and the problems of the working atmosphere and safety can be solved. Therefore, the titanium dioxide colloid sol and the method of producing the sol of the present invention are industrially valuable.

CLAIMS:

1. A titanium dioxide colloid sol comprising 50 to 100 parts by weight of titanium dioxide colloidal particles charged with negative electricity and 5 to 50 parts by weight of a complexing agent and 1 to 50 parts by weight of an alkaline substance, the complexing agent comprising at least one member selected from the group consisting of condensed phosphoric acids and condensed phosphate salts.
2. The titanium dioxide colloid sol as claimed in claim 1, wherein the pH value of the titanium dioxide sol is 5 to 10.
3. The titanium dioxide colloid sol as claimed in claim 1, wherein the alkaline substance comprises at least one member selected from the group consisting of ammonium compounds, alkali metal compounds and amine compounds.
4. The titanium dioxide colloid sol as claimed in claim 1, wherein the alkaline substance comprises at least one member selected from the group consisting of oxazine, piperidine and choline compounds.
5. A method of producing a titanium dioxide colloid sol characterized by mixing an alkaline substance into an acid titanium dioxide colloid sol containing 50 to 100 parts by weight of titanium dioxide colloid sol and 5 to 50 parts by weight of a complexing agent, to adjust the pH value of the sol to a level of 5 to 10, and to thereby cause the titanium dioxide colloidal particles to be charged with negative electricity, the complexing agent comprising at least one member selected from the group consisting of condensed phosphoric acid and condensed phosphate salts.
6. The method of producing the titanium dioxide colloid sol as claimed in claim 5,

wherein the alkaline substance comprises at least one member selected from the group consisting of ammonium compounds, alkali metal compounds and amine compounds.

7. The method of producing the titanium dioxide colloid sol as claimed in claim 5, wherein the alkaline substance comprises at least one member selected from the group consisting of oxazine, piperidine and choline compounds.

8. A method of producing al titanium dioxide colloid sol, characterized by mixing an alkaline substance into an acid titanium dioxide colloid sol comprising 50 to 100 parts by weight of titanium dioxide colloidal particles and 5 to 50 parts by weight of a complexing agent to adjust the pH value of the resultant sol to a level of 6 to 12, and applying a deionization treatment to the sol to thereby cause the titanium dioxide colloidal particles to be charged with negative electricity.

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L4: Entry 4 of 37

File: USPT

Jul 16, 2002

US-PAT-NO: 6420437

DOCUMENT-IDENTIFIER: US 6420437 B1

TITLE: Titanium oxide colloidal sol and process for the preparation thereof

DATE-ISSUED: July 16, 2002

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Mori; Kazuhiko	Tokyo			JP
Nakamura; Mitsuru	Tokyo			JP
Tanaka; Masanobu	Tokyo			JP

ASSIGNEE-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY	TYPE CODE
Nihon Parkerizing Co., Ltd.	Tokyo			JP	03

APPL-NO: 09/ 601034 [PALM]

DATE FILED: July 26, 2000

FOREIGN-APPL-PRIORITY-DATA:

COUNTRY	APPL-NO	APPL-DATE
JP	10-013811	January 27, 1998
JP	10-282938	October 5, 1998

PCT-DATA:

APPL-NO	DATE-FILED	PUB-NO	PUB-DATE	371-DATE	102(E)-DATE
PCT/JP99/00342	January 27, 1999	WO99/37582	Jul 29, 1999	Jul 26, 2000	Jul 26, 2000

INT-CL: [07] B01 F 17/14, B01 F 17/16, C09 C 1/36, C09 K 3/18, F21 V 9/06

US-CL-ISSUED: 516/90; 106/13, 106/287.19, 106/440, 106/448, 252/588, 502/350

US-CL-CURRENT: 516/90; 106/13, 106/287.19, 106/440, 106/448, 252/588, 502/350

FIELD-OF-SEARCH: 516/90, 106/287.19, 106/440, 106/448, 106/13, 502/350, 252/588

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

Search Selected

Search ALL

	PAT-NO	ISSUE-DATE	PATENTEE-NAME	US-CL
<input type="checkbox"/>	<u>2819177</u>	January 1958	Conn et al.	
<input type="checkbox"/>	<u>3147131</u>	September 1964	Kingsbury	106/448
<input type="checkbox"/>	<u>3567479</u>	March 1971	Portes et al.	106/448
<input type="checkbox"/>	<u>3582275</u>	June 1971	Sugahara et al.	
<input type="checkbox"/>	<u>3663284</u>	May 1972	Stancioff et al.	106/448 X
<input type="checkbox"/>	<u>4448609</u>	May 1984	Tear et al.	516/90 X
<input type="checkbox"/>	<u>4576921</u>	March 1986	Lane	516/90 X
<input type="checkbox"/>	<u>5049309</u>	September 1991	Sakamoto et al.	516/90
<input type="checkbox"/>	<u>5059248</u>	October 1991	Signorino et al.	516/90 X
<input type="checkbox"/>	<u>5698205</u>	December 1997	Bruckner et al.	516/90 X
<input type="checkbox"/>	<u>5759251</u>	June 1998	Nakamura et al.	106/287.19 X

FOREIGN PATENT DOCUMENTS

FOREIGN-PAT-NO	PUBN-DATE	COUNTRY	US-CL
1 271 862	July 1968	DE	106/448
0376216	July 1990	EP	
0684075	November 1995	EP	
7-89722	April 1995	JP	
10-18082	January 1998	JP	

OTHER PUBLICATIONS

Riskin et al. "Colloidal stability of titanium dioxide sols in concentrated hydrochloric Acid solutions" (1970), Chemical Abstracts No. 79:70516.

ART-UNIT: 1712

PRIMARY-EXAMINER: Lovering; Richard D.

ABSTRACT:

A neutral titanium dioxide colloid sol having a high stability in a neutral range and capable of forming a colorless transparent coating even when dried at room temperature is produced by mixing an acid titanium dioxide sol comprising 50 to 100 parts by weight of titanium dioxide colloidal particles and 5 to 50 parts by weight of a chelating agent for titanium ions with 1 to 50 parts by weight of an alkaline substance comprising at least one of alkali metal compounds and amine compounds, and optionally by adjusting the pH value of the liquid mixture to 5 to 10, or adjusting the pH value of the liquid mixture to 6 to 10, and then applying a deionization treatment to the mixture to thereby charge the titanium dioxide colloidal particles with negative electricity.

8 Claims, 0 Drawing figures

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L4: Entry 6 of 37

File: USPT

May 15, 2001

US-PAT-NO: 6231343

DOCUMENT-IDENTIFIER: US 6231343 B1

TITLE: Method for bleaching discolored tooth by titanium dioxide photocatalyst

DATE-ISSUED: May 15, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Ishibashi; Takuro	Nagasaki	817-001		JP
Ishibashi; Kouzo	Nagasaki	817-001		JP
Taoda; Hiroshi	Nagoya-shi			JP
Nonami; Toru	Nagoya-shi			JP

US-CL-CURRENT: 433/215; 433/216

CLAIMS:

What is claimed is:

1. A method for bleaching discolored teeth comprising applying a solution or paste or both comprising a titanium dioxide powder and hydrogen peroxide solution onto the surface of the discolored teeth, and irradiating the discolored teeth with light, thereby bleaching the teeth by resultant photocatalytic action.
2. The method for bleaching discolored teeth according to claim 1, wherein said irradiating light is violet or blue visible light.
3. The method for bleaching discolored teeth according to claim 1, which comprises:
 - a) cleaning surfaces of the discolored teeth;
 - b) moisture-proofing said cleaned surfaces; and
 - c) coating said moisture-proofed surfaces with said solution or paste or both of said titanium dioxide powder and said hydrogen peroxide, and irradiating said coated surfaces with visible light.
4. The method for bleaching discolored teeth according to claim 3, wherein step c) is repeated, whereby fresh paste or solution or both is applied and irradiated every 15 to 20 minutes.
5. The method for bleaching discolored teeth according to claim 4, wherein step c) is repeated 2 or 3 times.
6. The method for bleaching discolored teeth according to claim 5, wherein step c) is repeated 4 or 5 times.
7. The method for bleaching discolored teeth according to claim 1, wherein said solution or past further comprises trace amounts of iron salts.
8. A bleaching composition for bleaching discolored teeth by photocatalytic

action produced by irradiation with light, wherein

said bleaching composition comprises as active ingredients a combination of 6% or less hydrogen peroxide solution and crystalline titanium dioxide photocatalyst, wherein

said photocatalyst produces the photocatalytic action and reacts with the hydrogen peroxide to produce active oxygen when irradiated with light.

9. The bleaching composition according to claim 8, which consists essentially of a solution/paste of titanium dioxide with a particle diameter of about 5 to 60 nm and 3% or less hydrogen peroxide solution.

10. The bleaching composition according to claim 8, which further comprises trace amounts of iron salts.

11. A method for producing a bleaching composition for bleaching discolored teeth by photocatalytic action produced by irradiation with light, which method comprises:

blending 6% or less hydrogen peroxide solution with a crystalline titanium dioxide photocatalyst powder, wherein

said photocatalyst producing the photocatalytic action and reacts with hydrogen peroxide to produce active oxygen when irradiated with light.

12. The method for producing a bleaching composition according to claim 11, wherein 3% or less hydrogen peroxide solution is blended with the crystalline titanium dioxide photocatalyst powder.

13. The method for producing a bleaching agent according to claim 11, wherein the crystalline titanium dioxide photocatalyst powder is anatase-type titanium dioxide.

14. A system for bleaching teeth, comprising a combination of a bleaching composition, materials or device for applying said bleaching composition, irradiating device, and optionally other dental treatment materials, wherein the bleaching composition comprises as active ingredients a combination of 6% or less hydrogen peroxide solution and crystalline titanium dioxide photocatalyst,

said photocatalyst producing the photocatalytic action and reacting with the hydrogen peroxide to produce active oxygen when irradiated with light.

15. The system for bleaching teeth according to claim 14, wherein the radiating device generates visible violet light.

16. The system for bleaching teeth according to claim 15, wherein the radiating device generates LED violet light.

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L4: Entry 6 of 37

File: USPT

May 15, 2001

US-PAT-NO: 6231343

DOCUMENT-IDENTIFIER: US 6231343 B1

TITLE: Method for bleaching discolored tooth by titanium dioxide photocatalyst

DATE-ISSUED: May 15, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Ishibashi; Takuro	Nagasaki	817-001		JP
Ishibashi; Kouzo	Nagasaki	817-001		JP
Taoda; Hiroshi	Nagoya-shi			JP
Nonami; Toru	Nagoya-shi			JP

ASSIGNEE-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY	TYPE	CODE
Agency of Industrial Science and Technology	Tokyo			JP		07
Ishibashi; Takuro	Shimoagata-gun			JP		05
Ishibashi; Kouzo	Shimoagata-gun			JP		05
Taoda; Hiroshi	Nagoya			JP		05
Nonami; Toru	Nagoya			JP		05

APPL-NO: 09/ 508109 [PALM]

DATE FILED: June 9, 2000

FOREIGN-APPL-PRIORITY-DATA:

COUNTRY	APPL-NO	APPL-DATE
JP	9-273650	September 19, 1997

PCT-DATA:

APPL-NO	DATE-FILED	PUB-NO	PUB-DATE	371-DATE	102(E)-DATE
PCT/JP98/04220	September 18, 1998	WO99/15143	Apr 1, 1999	Jun 9, 2000	Jun 9, 2000

INT-CL: [07] A61 C 5/00

US-CL-ISSUED: 433/215; 433/216

US-CL-CURRENT: 433/215; 433/216

FIELD-OF-SEARCH: 433/215, 433/216, 433/229, 433/80

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

Search Selected

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	PAT-NO	ISSUE-DATE	PATENTEE-NAME	US-CL
<input type="checkbox"/>	<u>5766574</u>	June 1998	Christina-Beck et al.	424/53
<input type="checkbox"/>	<u>5902568</u>	May 1999	Ryles et al.	424/53
<input type="checkbox"/>	<u>5914305</u>	June 1999	Madison et al.	510/367

ART-UNIT: 372

PRIMARY-EXAMINER: Lucchesi; Nicholas D.

ABSTRACT:

The present invention relates to a method for bleaching a discolored tooth, comprising the steps of applying a solution/paste of titanium dioxide powder and hydrogen peroxide solution onto the surface of discolored teeth, and bleaching the tooth based on the photocatalytic action that is produced by irradiating this area with light; and to a bleaching agent for applying onto the surface of discolored tooth to bleach the discolored tooth based on the photocatalytic action that is produced by irradiating this area with light, the bleaching agent comprising as active ingredients a combination of hydrogen peroxide solution and titanium dioxide producing photocatalytic action when irradiated with light.

16 Claims, 0 Drawing figures

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L4: Entry 6 of 37

File: USPT

May 15, 2001

DOCUMENT-IDENTIFIER: US 6231343 B1

TITLE: Method for bleaching discolored tooth by titanium dioxide photocatalystAbstract Text (1):

The present invention relates to a method for bleaching a discolored tooth, comprising the steps of applying a solution/paste of titanium dioxide powder and hydrogen peroxide solution onto the surface of discolored teeth, and bleaching the tooth based on the photocatalytic action that is produced by irradiating this area with light; and to a bleaching agent for applying onto the surface of discolored tooth to bleach the discolored tooth based on the photocatalytic action that is produced by irradiating this area with light, the bleaching agent comprising as active ingredients a combination of hydrogen peroxide solution and titanium dioxide producing photocatalytic action when irradiated with light.

Brief Summary Text (3):

The present invention relates to the utilization of photocatalytic action to bleach and remove colored sediment on (stained or discolored) teeth, and more particularly to a method for bleaching discolored teeth comprising the steps of applying a bleaching agent comprising a specific composition having photocatalytic action on the surface of discolored teeth, and bleaching the teeth based on the photocatalytic action that is produced by irradiating this area with light; a novel bleaching agent comprising a solution/paste of 3% or less aqueous hydrogen peroxide and titanium dioxide producing photocatalytic action when irradiated with light, which is useful for such bleaching; a method for producing such a bleaching agent; and a bleaching system comprising a combination of the aforementioned bleaching agent and the like.

Brief Summary Text (5):

There has been increasing demand in recent years for improvements in the contour, alignment, and integrity of teeth in dental therapy, which may be referred to as cosmetic improvements. Recently there have been more cases of patients seeking dental therapy based on a desire for whiter teeth, which is increasingly regarded by young women as an important element of beauty. The causes of dental discoloration and pigmentation or staining are generally classified into so-called extrinsic factors, such as the sedimentation of colored substances (tobacco, tea, etc.), pigment generating bacteria, the discoloration of repair materials (primarily composite resins), and metal salts (primarily amalgams, silver nitrate, and ammonia silver), and intrinsic factors, such as aging, chemicals or drugs (such as fluorine and tetracyclines), dysmetabolism and hereditary diseases, and dental injuries. The latter intrinsically discolored teeth are indicated for bleaching methods to which dental health system treatment is applied.

Brief Summary Text (6):

Several methods have been proposed in the past as methods for the cosmetic improvement of discolored teeth, among which bleaching may be considered a highly effective method for the preservation of dentine, despite drawbacks such as occasional recurrence, when methods suited to individual cases are selected and properly carried out.

Brief Summary Text (9):

1) Bleaching which features the use of 30% H.sub.2 O.sub.2 as the chemical agent, and the combined use of light and heat on the aqueous hydrogen peroxide.

Brief Summary Text (10):

Brief Summary Text (11):

Brief Summary Text (12):

Brief Summary Text (13):

Brief Summary Text (14):

Brief Summary Text (16) :

Brief Summary Text (18) :

Brief Summary Text (21) :

Brief Summary Text (22) :

The following conditions are required of bleaching methods and bleaching agents during dental bleaching, however:

Brief Summary Text (26):

(d) avoidance of detracting from dental physical properties after treatment;

Brief Summary Text (29):

A bleaching method fulfilling the above conditions would be capable of affording cosmetic improvement while preserving dental contour, with considerably improved effects.

Brief Summary Text (30):

In conventional bleaching methods, however, the primary chemical agent is 30 to 35% aqueous hydrogen peroxide, which is highly corrosive, and the oxidative action of which is the basis of bleaching.

Brief Summary Text (31):

All of the various bleaching methods currently used in Japan can be said to comprise, as noted above, a combination of 30 to 35% aqueous hydrogen peroxide, various utensils, and other chemical agents. There is an example of a bleaching method employed in the United States which features the use of 10% urea peroxide instead of 30 to 35% aqueous hydrogen peroxide, but this method is currently embroiled in controversy over problems in terms of efficacy and safety, and has yet to gain approval in Japan.

Brief Summary Text (32):

Bleaching methods covered as insured treatment are currently limited to pulpless teeth, and in some cases pulpectomy is performed on non-carious teeth for the purpose of bleaching. In addition, the use of highly toxic 30 to 35% aqueous hydrogen peroxide in various bleaching methods restricts operations and the like in a variety of ways, while limited bleaching effects for pulped teeth in particular have been indicated.

Brief Summary Text (33):

There is thus a strong need for the development of a safer, faster, simpler novel dental bleaching method that would be effective for both vital teeth and pulpless teeth.

Brief Summary Text (35):

The present invention is intended to provide a method for bleaching discolored teeth using a titanium dioxide photocatalyst.

Brief Summary Text (36):

The present invention relates to a method for bleaching discolored teeth, comprising the steps of applying a solution/paste of titanium dioxide powder and aqueous hydrogen peroxide to the surface of discolored teeth, and bleaching the teeth based on the photocatalytic action that is produced by irradiating this area with light; and also to a bleaching agent which is applied to the surface of discolored teeth to bleach the discolored teeth based on the photocatalytic action that is produced by irradiating this area with light, the aforementioned bleaching agent comprising as active ingredients a combination of aqueous hydrogen peroxide and titanium dioxide producing photocatalytic action when irradiated with light.

Brief Summary Text (37):

Characteristic effects afforded by the present invention include the following: (1) the ability to provide a novel bleaching agent for discolored teeth; (2) the ability to bleach both vital teeth and pulpless teeth; (3) higher safety, better workability, and rapid bleaching effects; (4) alleviation of mental distress on treated patients; (5) the ability to control the level of color adaptation; and (6) the ability to select improvements in color adaptation desired by the treated patient.

Brief Summary Text (39):

As a result of extensive research in light of the foregoing to establish a novel bleaching method that would be safer and simpler, and that would also afford better bleaching effects, the inventors perfected the present invention upon discovering that the intended objectives can be achieved by the combined use of active

ingredients comprising aqueous hydrogen peroxide and titanium dioxide having photocatalytic action.

Brief Summary Text (40):

That is, an object of the present invention is to provide a safer, faster, simpler novel dental bleaching method that would be effective for both vital teeth and pulpless teeth.

Brief Summary Text (41):

A further object of the present invention is to provide a novel bleaching agent used in the aforementioned bleaching method, a method for its manufacture, and a system for bleaching teeth using the aforementioned bleaching agent.

Brief Summary Text (43):

(1) A method for bleaching a discolored tooth, comprising the steps of applying a solution/paste of a titanium dioxide powder and hydrogen peroxide solution onto the surface of the discolored tooth, and irradiating this area with light to bleach the tooth based on the resultant photocatalytic action.

Brief Summary Text (44):

(2) The method for bleaching a discolored tooth according to (1) above, wherein violet or blue visible light is irradiated.

Brief Summary Text (45):

(3) A bleaching agent for applying onto a surface of a discolored tooth to bleach the discolored tooth based on a photocatalytic action that is produced by irradiating this area with light, said bleaching agent comprising as active ingredients a combination of hydrogen peroxide solution and titanium dioxide which produces the photocatalytic action when irradiated with light.

Brief Summary Text (46):

(4) The bleaching agent according to (3) above, consisting of a solution/paste of titanium dioxide with a particle diameter of about 5 to 60 nm and 3% or less hydrogen peroxide solution.

Brief Summary Text (47):

(5) A method for producing a bleaching agent for applying onto a surface of a discolored tooth to bleach the discolored teeth based on the photocatalytic action that is produced by irradiating this area with light, said method for producing a bleaching agent comprising the step of blending hydrogen peroxide solution with a titanium dioxide powder producing photocatalytic action when irradiated with light.

Brief Summary Text (48):

(6) The method for producing a bleaching agent according to (5) above, comprising blending 3% or less hydrogen peroxide solution with a titanium dioxide powder producing photocatalytic action when irradiated with light.

Brief Summary Text (50):

(8) A system for bleaching tooth comprising a combination of a bleaching agent according to (3) or (4) above, means for applying said bleaching agent, irradiating means, and/or other dental treatment materials.

Brief Summary Text (51):

(9) The system for bleaching tooth according to (8) above, wherein a radiation device for generating visible violet light is combined.

Brief Summary Text (53):

To achieve the objects of the present invention, the present invention provides high bleaching effects for discolored teeth, primarily through the oxidative and reductive action of the photocatalyst. In the present invention, the term discoloration is defined as expressing a broad range of meanings including staining.

Brief Summary Text (54):

The chemical agents and utensils used in the present invention may essentially

comprise titanium oxide powder, aqueous hydrogen peroxide, and visible light rays (radiating instrument), affording considerable stability, ease of operation, and bleaching effects. The aforementioned aqueous hydrogen peroxide is more suitably used in lower concentrations, such as a concentration of 6% or less, and preferably a lower concentration of 3% or less.

Brief Summary Text (55):

In a preferred embodiment, the bleaching agent of the present invention is composed of a solution/paste of titanium dioxide powder and aqueous hydrogen peroxide in a low concentration of, for example, 6% or less (and preferably 3% or less). Examples of desirable titanium dioxide include, but are not limited to, titanium dioxide having a particle diameter of about 5 to 60 nm, and particularly anatase type titanium dioxide fine particles or materials based thereon. Any titanium dioxide having similar effects, that is, producing photocatalytic action, can be used, no matter what the shape or properties. Advantages of a smaller particle diameter in this case are that higher activity can be anticipated, good effects can be obtained with lower amounts, lower amounts can be used, high bleaching effects can be obtained more rapidly because thinner coating films can be used, for example, and so forth.

Brief Summary Text (56):

The weight ratio of the aforementioned ingredients can be adjusted as desired depending, for example, on whether the degree of discoloration is lighter or greater, so that products suited to individual cases can be prepared. The bleaching agent of the present invention is ordinarily, but not only, used in the form of a uniform transparent solution or paste by blending, kneading, and dispersing the titanium dioxide powder in aqueous hydrogen peroxide of low concentration. Any type that is prepared in a manner similar to this is encompassed within the range of the present invention. The term "solution/paste" as used in the present invention is defined as any having the aforementioned meaning. In this case, the means and devices for preparing the bleaching agent such as by blending, kneading, dispersion or otherwise manipulating the aforementioned ingredients, as well as the means and the like for applying the bleaching agent, are not particularly limited. Any suitable type can be used. An example of a desirable method for applying the bleaching agent to dental surfaces is to directly coat the dental surface with the bleaching agent.

Brief Summary Text (57):

At this time, fabric, paper, glass cloth, ceramic paper, an organic gel, an inorganic gel, or the like can be impregnated with the bleaching agent of the present invention, that is, a solution or paste of the aqueous hydrogen peroxide and titanium dioxide having photocatalytic action, and the impregnated product can be applied to the dental surface and irradiated with light. Other suitable methods and means can also be used, such as a method in which the aforementioned bleaching agent is supported on a suitable carrier, and is fitted or applied to a tooth or row of teeth.

Brief Summary Text (59):

Discolored teeth can be bleached with the aforementioned bleaching agent by repeatedly applying a solution or paste of a titanium dioxide powder and 3% or less aqueous hydrogen peroxide to a dental surface, for example, and irradiating the coated area. The number of times the material is applied and irradiated may be suitably adjusted according to how light or heavy the staining is. The aforementioned solution or paste should usually be applied by coating or the like using fresh solution or paste about every 15 to 20 minutes. The interval and frequency should be suitably determined according to the state of the teeth.

Brief Summary Text (60):

The bleaching agent of the present invention is effective for bleaching both pulpless teeth and vital teeth, and produces pronounced effects while allowing the teeth to be safely and easily bleached.

Brief Summary Text (61):

The primary action of the bleaching agent in the present invention is bleaching action based on the synergistic action of the titanium dioxide photocatalyst and low

concentration aqueous hydrogen peroxide (for example, 6% or less, and preferably 3% or less, aqueous hydrogen peroxide).

Brief Summary Text (62):

When the titanium dioxide photocatalyst is irradiated with light, electrons and positive holes are produced, reacting with hydrogen peroxide to produce active oxygen. The active oxygen has far greater oxidizing power than ozone, and can oxidize nearly all organic materials into carbon dioxide. Even when n type semiconductor titanium powder with a relatively substantial band gap is used in the form of a solution with 3% aqueous hydrogen peroxide, for example, light radiation readily results in the production of active oxygen having potent oxidizing power, ensuring higher levels of charge separation, electron hole mobility, reactivity with protons or hydroxyl groups, or the like than when used alone, so that synergistic action is produced in addition to the oxidizing action of the 3% aqueous hydrogen peroxide itself.

Brief Summary Text (64):

Dental staining factors are broadly classified into the following extrinsic and intrinsic factors.

Brief Summary Text (75):

II. Bleeding in dental pulp (from external trauma, following pulpectomy, arsenious acid)

Brief Summary Text (77):

IV. Tooth decay, rheumatic fever

Brief Summary Text (86):

The aforementioned staining factors are caused by a variety of pigments, iron salts, tannic acid, chlorhexidine, benzalkonium chloride-chlorhexidine, and cyclones. Such colored substances adhere to tooth enamel and dentine.

Brief Summary Text (87):

A solution of titanium oxide and, for example, 6% or less, and preferably 3% or less, aqueous hydrogen peroxide penetrates between the enamel prisms and dentine, and bleaching is brought about as colored substances are degraded by the oxidative and reductive action of the photocatalyst. The bleaching method of the present invention affords high bleaching effects for discolored teeth caused by both intrinsic and extrinsic factors.

Brief Summary Text (89):

In the present invention, the aforementioned bleaching agent, means for applying the bleaching agent (applicators, etc.), radiation instruments, other chemical agents, other dental treatment materials, instruments, and the like can be suitably combined to produce a system (kit) for bleaching teeth.

Detailed Description Text (3):

0.3 g of anatase type TiO_2 powder with a particle diameter of 7 nm was blended with 1 mL each of a) 0.5% aqueous hydrogen peroxide, b) 1.5% aqueous hydrogen peroxide, and c) 3% aqueous hydrogen peroxide, and the ingredients were kneaded and dispersed to prepare solutions of titanium dioxide and aqueous hydrogen peroxide. 1 g of anatase type TiO_2 powder with a particle diameter of 7 nm was similarly used to prepare a d) paste of 3% aqueous hydrogen peroxide. The products were then housed in containers shielded from light, giving bleaching agents.

Detailed Description Text (4):

(2) Preparation of System for Bleaching Teeth

Detailed Description Text (5):

The aforementioned bleaching agents were housed in containers together with an applicator, radiating instrument (using LED violet light), and preparatory utensils, so as to prepare a system (kit) for bleaching teeth.

Detailed Description Text (6):

(3) Bleaching Discolored Teeth

Detailed Description Text (7):

The aforementioned bleaching agents were used to bleach discolored teeth by the following procedure.

Detailed Description Text (9):

2) The surfaces of the teeth were cleaned by a common method using a rubber cap or the like, and were then dried.

Detailed Description Text (10):

3) The teeth were then temporarily moisture-proofed.

Detailed Description Text (11):

4) The dental surface was coated with a solution of titanium oxide and 3% aqueous hydrogen peroxide, and irradiated with visible light.

Detailed Description Text (15):

Table 1 shows the results obtained with the aforementioned bleaching agents. It is apparent from Table 1 that teeth with mild discoloration (F1) enjoyed considerable bleaching effects after about 1 procedure described above, and that teeth with moderate discoloration (F2 to F3) enjoyed considerable bleaching effects after about 2 to 3 times. Teeth with pronounced discoloration (F4) were bleached after about 4 to 5 times. The aforementioned bleaching effects were long-lasting, with no need for return treatment.

Detailed Description Text (16):

Since the bleaching agent of the present invention provides excellent bleaching effects through the synergism between the bleaching action based on the titanium oxide photocatalyst and the bleaching action of the aqueous hydrogen peroxide, there are no operational restrictions such as those in the case of conventional, highly toxic 30 to 35% aqueous hydrogen peroxide. It may also be understood that the bleaching agent of the present invention can be used for both pulped and pulpless teeth, since it is highly safe.

Detailed Description Text (17):

Compared, in terms of oxidizing energy, to conventional bleaching agents based on aqueous hydrogen peroxide, the bleaching agent in the present invention can be seen to have rapidly resulted in about 2.9 times or more greater bleaching effects.

Detailed Description Text (18):

It is also evident from Table 1 that lower aqueous hydrogen peroxide concentrations tended to require a longer period of time.

Detailed Description Text (19):

The bleaching effects were also enhanced, irrespective of H.sub.2 O.sub.2 concentration, by mixing a solution containing trace amounts of iron salts with the aforementioned bleaching agent applied to the teeth, and then irradiating the coated portion.

Detailed Description Text (30):

(1) Bleaching of Discolored Teeth by Application of Bleaching Agent and Irradiation

Detailed Description Text (31):

Dental surfaces (of extracted teeth) were coated with a bleaching agent (solution) comprising 0.5 g TiO₂ powder mixed with 3% H₂O₂, and were irradiated with visible light. The coating and irradiation were repeated in the same manner as in Example 1.

Detailed Description Text (32):

As a control, discolored teeth (extracted teeth) were coated with 3% H₂O₂ by itself, and irradiated with visible light and UV rays continuously for 10 hours.

Detailed Description Text (34):

Following common dental surface cleaning, attempts were made to bleach discolored teeth by applying and irradiating an agent according to a) through c) below.

Detailed Description Text (35):

a) The surface of teeth were coated with the aforementioned TiO_2 -3% H_2O_2 solution using an applicator, and then irradiated with visible light.

Detailed Description Text (36):

b) Dental surfaces were covered with paper ceramic containing TiO_2 powder (by Noritake Company Ltd.). 3% H_2O_2 was then applied thereon using an applicator, and the material was irradiated.

Detailed Description Text (37):

c) Dental surfaces were coated with a paste comprising TiO_2 -3% H_2O_2 (1.2 g/mL) as active ingredients, and were irradiated.

Detailed Description Text (38):

(3) Concentration of Aqueous Hydrogen Peroxide

Detailed Description Text (39):

The concentration of aqueous hydrogen peroxide was varied within the range from 0.1% to 35%, and attempts were made to bleach discolored teeth by applying and irradiating (visible light) the material in the same manner as above.

Detailed Description Text (40):

Extremely discolored teeth were coated on the surface with 30 to 35% H_2O_2 -- TiO_2 solutions and irradiated with UV rays.

Detailed Description Text (43):

The type of radiated light (wavelength of 250 to 600 nm) was varied in attempts to bleach discolored teeth by applying and irradiating the material in the same manner as above.

Detailed Description Text (45):

Considerable bleaching effects were noted after about 2 hours in (1) above. Except for discolored teeth resulting from pulp necropathy, the bleaching effects as a whole were excellent. Excellent bleaching effects were also obtained by adjusting the irradiation time according to the degree of discoloration. It was also possible to control adjustments to suitable levels by adjusting the method and time. No bleaching effects were noted in controls.

Detailed Description Text (46):

There were no significant differences in bleaching effects due to bleaching agent configuration or method in (2) above. Technicians can accordingly make their selections by determining the workability depending on the site of the discolored teeth.

Detailed Description Text (50):

As described above, the present invention relates to a bleaching agent for bleaching discolored teeth based on the photocatalytic action that is produced when the agent is applied to the surface of discolored teeth and is irradiated, comprising as active ingredients a combination of aqueous hydrogen peroxide and titanium dioxide producing photocatalytic action when irradiated. The present invention is extremely useful for cosmetic improvements of teeth because of the following effects: (1) the ability to provide a novel bleaching agent for discolored teeth; (2) the ability to bleach both vital teeth and pulpless teeth; (3) higher safety, better workability, and rapid bleaching effects; (4) alleviation of mental distress on treated patients; (5) the ability to control the level of color adaptation; and (6) the ability to select improvements in color adaptation desired by the treated patient.

CLAIMS:

1. A method for bleaching discolored teeth comprising applying a solution or paste or both comprising a titanium dioxide powder and hydrogen peroxide solution onto the surface of the discolored teeth, and irradiating the discolored teeth with light, thereby bleaching the teeth by resultant photocatalytic action.

2. The method for bleaching discolored teeth according to claim 1, wherein said irradiating light is violet or blue visible light.
3. The method for bleaching discolored teeth according to claim 1, which comprises:
 - a) cleaning surfaces of the discolored teeth;
 - b) moisture-proofing said cleaned surfaces; and
 - c) coating said moisture-proofed surfaces with said solution or paste or both of said titanium dioxide powder and said hydrogen peroxide, and irradiating said coated surfaces with visible light.
4. The method for bleaching discolored teeth according to claim 3, wherein step c) is repeated, whereby fresh paste or solution or both is applied and irradiated every 15 to 20 minutes.
5. The method for bleaching discolored teeth according to claim 4, wherein step c) is repeated 2 or 3 times.
6. The method for bleaching discolored teeth according to claim 5, wherein step c) is repeated 4 or 5 times.
7. The method for bleaching discolored teeth according to claim 1, wherein said solution or past further comprises trace amounts of iron salts.
8. A bleaching composition for bleaching discolored teeth by photocatalytic action produced by irradiation with light, wherein

said bleaching composition comprises as active ingredients a combination of 6% or less hydrogen peroxide solution and crystalline titanium dioxide photocatalyst, wherein

said photocatalyst produces the photocatalytic action and reacts with the hydrogen peroxide to produce active oxygen when irradiated with light.
9. The bleaching composition according to claim 8, which consists essentially of a solution/paste of titanium dioxide with a particle diameter of about 5 to 60 nm and 3% or less hydrogen peroxide solution.
11. A method for producing a bleaching composition for bleaching discolored teeth by photocatalytic action produced by irradiation with light, which method comprises:

blending 6% or less hydrogen peroxide solution with a crystalline titanium dioxide photocatalyst powder, wherein

said photocatalyst producing the photocatalytic action and reacts with hydrogen peroxide to produce active oxygen when irradiated with light.
12. The method for producing a bleaching composition according to claim 11, wherein 3% or less hydrogen peroxide solution is blended with the crystalline titanium dioxide photocatalyst powder.
13. The method for producing a bleaching agent according to claim 11, wherein the crystalline titanium dioxide photocatalyst powder is anatase-type titanium dioxide.
14. A system for bleaching teeth, comprising a combination of a bleaching composition, materials or device for applying said bleaching composition, irradiating device, and optionally other dental treatment materials, wherein the bleaching composition comprises as active ingredients a combination of 6% or less hydrogen peroxide solution and crystalline titanium dioxide photocatalyst,

said photocatalyst producing the photocatalytic action and reacting with the hydrogen peroxide to produce active oxygen when irradiated with light.

15. The system for bleaching teeth according to claim 14, wherein the radiating device generates visible violet light.
16. The system for bleaching teeth according to claim 15, wherein the radiating device generates LED violet light.

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L4: Entry 7 of 37

File: USPT

Apr 3, 2001

US-PAT-NO: 6210163

DOCUMENT-IDENTIFIER: US 6210163 B1

TITLE: Composition and method for cosmetically improving and altering the appearance of teeth

DATE-ISSUED: April 3, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Cohen; Morton	Elkins Park	PA	19027	

US-CL-CURRENT: 433/217.1; 106/35

CLAIMS:

What is claimed is:

1. A method for cosmetically altering the appearance of teeth to whiten or colorize the teeth, comprising the steps of:

a) preparing the tooth by exposing the tooth so that the tooth surface to be altered is visible to the gum line;

b) preparing a lac based covering compound to be applied onto the enamel surface of the tooth to be altered, including selecting a compound from a plurality of tooth colored compounds to match the shade of the adjacent or surrounding teeth in the individual's mouth;

c) applying the covering compound prepared in step b. to the tooth surface to be coated; and

d) allowing the covering compound applied to the tooth to dry by exposing the tooth to air.

2. The method of claim 1, wherein the step of preparing a lac based covering compound includes selecting a color from one or more non-tooth colored compounds and mixing said non-tooth-colored compound with said lac based covering compound to provide an overall non-tooth colored appearance, and wherein the covering compound is applied to the tooth surface evenly to cover the tooth surface and is permitted to cure by drying the compound to provide a non-tooth colored coating on the surface of the tooth.

3. The method of claim 2, further including the step of providing palette means, said palette means including a plurality of pigmented compounds, and selecting one or more of said pigmented compounds from said palette means and mixing said selected one or more pigmented compounds with said the lac based covering compound of step b) before applying said lac based covering compound to said tooth.

4. The method of claim 2, further including the step of providing a food grade dye as the colorizing compound and blending said food grade dye with said lac based covering compound to provide a colorized covering compound.

5. The method of claim 1, further including the step of selectively removing the lac based covering compound applied to the tooth.
6. The method of claim 5, wherein the step of selectively removing the lac based covering compound from the tooth includes contacting the compound with a solvent.
7. The method of claim 5, wherein the step of selectively removing the lac based covering compound includes applying to the tooth surface containing the lac based covering compound a forced stream of water.
8. The method of claim 1, further comprising the step of providing colorizing means containing a plurality of pigments to form palette means, and wherein the method further includes the steps of selecting at least one pigment from said palette means, and mixing said selected pigment with said lac based covering compound to be applied to the tooth.
9. The method of claim 1, further including the step of mixing a reflecting material into said lac based covering compound to be applied to said tooth.
10. The method of claim 1, wherein the step of preparing a lac based covering compound includes providing a lac resin and dissolving said lac resin in an ethanol solution.
11. The method of claim 1, wherein the step of preparing the lac based covering compound includes admixing pulverized fluoroaluminosilicate glass particles.
12. The method of claim 1, wherein the step of preparing a tooth further includes the steps of providing a weak acid solution and contacting the tooth surface with the weak acid solution.
13. The method of claim 12, wherein the step of contacting the tooth with a weak acid solution, includes etching the tooth with an acid selected from the group of citric acid and polyacrylic acid.
14. The method of claim 1, wherein the step of preparing a tooth further includes the step of etching microscopic pits which are less than about 5.0 .mu.m in the tooth surface with an etchant.
15. The method of claim 1, wherein the step of preparing a lac based covering compound further includes selecting a food grade dye and blending said dye with said covering compound to provide a colored covering on said tooth.
16. The method of claim 1, wherein the step of preparing a lac based covering compound further comprises providing said lac based covering compound in a fluidic suspension for delivery by spraying, and wherein the step of applying the covering compound to the tooth surface comprises the step of spraying the covering compound on the tooth surface.
17. A method for cosmetically altering the appearance of teeth to whiten or colorize the teeth, comprising the steps of:
 - a) preparing the tooth by exposing the tooth so that the tooth surface to be altered is visible to the gum line;
 - b) providing palette means containing a plurality of tooth-colored shaded pigment compounds, including the step of providing a container means for holding the pigment compounds and providing a mixing area;
 - c) selecting from said palette means one or more of said pigments contained in the container means to match the shade of adjacent or surrounding teeth in an individual's mouth;
 - d) preparing a lac based covering compound to be applied onto the enamel surface of the tooth to be altered by placing said lac based covering compound in said

mixing area of said container means and adding to said lac based covering compound from said container means a pigment compound from said palette means;

e) mixing said covering compound with said pigment compound;

f) applying the lac based covering compound prepared in step e) to the tooth surface to be coated; and

g) drying the covering compound applied to the tooth in step f) by exposing the covered tooth to air.

18. The method of claim 17, wherein the step of preparing a tooth further comprises the steps of:

a) providing a weak acid solution;

b) etching the tooth by contacting the tooth with a weak acidic solution to provide small microscopic pits in the enamel surface of said tooth; and

c) rinsing the weak acid solution off of the tooth with water.

19. A system for cosmetically altering the appearance of teeth wherein a removable coating is applied directly on a dry or wet tooth and cured on the tooth surface, comprising a lac based covering compound having a lac compound and a solvent, one or more colorizing pigment compounds, means for storing the lac based covering compound and associated means for storing one or more colorizing pigment compounds, wherein the lac based covering compound further comprises a compound selected from the group consisting of Bis-GMA sealants and glass ionomeric cements, wherein the system provides the user with the ability to select from the associated means one or more colorizing pigment compounds and mix said selected one or more colorizing pigment compounds with the lac based covering compound and to form a pigmented covering compound, apply the pigmented covering compound to cover the surface of the tooth to be altered, permit the pigmented covering compound to dry by exposure to air, and remove the pigmented covering compound from the tooth at the user's discretion.

20. A composition for cosmetically altering the appearance of teeth to whiten or colorize the teeth by applying said composition to a tooth surface, said composition comprising:

a) a lac resin;

b) a solvent for dissolving said lac resin; and

c) a pigment;

d) wherein the concentration of said lac resin is approximately 1 g/10 ml solvent, and wherein the lac resin is present in the composition in an amount which is greater than the amount of pigment in the composition.

21. The composition of claim 20, wherein said solvent comprises ethanol.

22. The composition of claim 21, wherein said lac resin comprises refined bleached food grade dewaxed lac.

23. The composition of claim 22, wherein said ethanol comprises specially denatured alcohol, formula 45/200 proof.

24. The composition of claim 23, wherein the concentration of said lac resin is approximately 34 to 37% by weight.

25. The composition of claim 20, wherein said pigment comprises titanium dioxide.

26. The composition of claim 20, further including a brightening agent.

27. The composition of claim 26, wherein said brightening agent comprises finely divided fluoroaluminosilicate compound.

28. A covering composition for application to the surface of a tooth for cosmetically altering the appearance of the tooth to whiten or colorize the tooth, said covering composition comprising dewaxed orange flake lac resin solubilized in an ethanol solution at a concentration of 1 g. per 10 ml of ethanol, and a pigment.

29. The covering compound of claim 28, wherein said ethanol solution comprises a 90% ethanol in water.

30. The covering compound of claim 28, wherein said pigment comprises titanium dioxide.

31. The covering compound of claim 28, wherein said pigment comprises a non-tooth colored shade for cosmetically colorizing the tooth when the covering compound is applied thereon.

32. A method for cosmetically altering the appearance of teeth to whiten or colorize the teeth, comprising the steps of:

a) preparing the tooth by exposing the tooth so that the tooth surface to be altered is visible to the gum line;

b) preparing a lac based covering compound to be applied onto the enamel surface of the tooth to be altered, including selecting a compound from a plurality of tooth colored compounds to match the shade of the adjacent or surrounding teeth in the individual's mouth;

c) applying the covering compound prepared in step b) to the tooth surface to be coated;

d) allowing the covering compound applied to the tooth to dry by exposing the tooth to air;

e) wherein the step of preparing a tooth further includes the the steps of providing a weak acid solution and contacting the tooth surface with the weak acid solution; and

f) wherein the weak acid solution is a citric acid of lemon juice.

33. A composition for cosmetically altering the appearance of teeth to whiten or colorize the teeth by applying said composition to a tooth surface, said composition comprising:

a) a lac resin;

b) a solvent for dissolving said lac resin;

c) a pigment;

d) wherein said solvent comprises ethanol; and

e) wherein said lac resin comprises refined bleached food grade dewaxed lac.

34. The composition of claim 33, wherein said ethanol comprises specially denatured alcohol, formula 45/200 proof.

35. The composition of claim 34, wherein the concentration of said lac resin is approximately 34% to 37% by weight.

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File: USPT

Apr 3, 2001

DOCUMENT-IDENTIFIER: US 6210163 B1

TITLE: Composition and method for cosmetically improving and altering the appearance of teethAbstract Text (1):

A composition and method for altering the appearance of teeth, including whitening or otherwise colorizing teeth, to cover discolorations and stains, or to provide a fashionable color on the tooth which can be selectively removed, the composition comprising a lac based compound solubilized for applying on the surface of a tooth, and a method including selecting the color to be applied, preparing the colorized compound to be applied to a tooth, exposing the tooth to be covered, applying a colorized compound to the enamel surface of the tooth, and allowing the compound to dry on the tooth, and selectively removing the compound from the tooth.

Brief Summary Text (3):

The present invention relates to the field of cosmetically improving and altering the appearance of teeth.

Brief Summary Text (5):

Many procedures are done to improve the appearance of teeth. Teeth are filled to replace dentin and enamel invaded by bacteria, and can be capped to replicate a removed or abraded portion of a tooth.

Brief Summary Text (6):

The teeth of individuals widely vary in their appearance and shape. This is due to the genetic make-up of the individual, but can also be affected by age, and the degree of contact with various foods and medications, both those superficially contacting the teeth and from the internal effects of the medication. The teeth of some individuals exhibit a yellow appearance while those of others may be whiter. While aging is often considered a "natural" cause of tooth discoloration, other factors commonly attributed to tooth discoloration can include chemical exposure to tannins, which are found in red wines, and brewed beverages such as coffees and teas. Additional discoloring chemicals include those not naturally occurring in foods, but rather, manufactured or synthesized compounds, such as, for example, the compounds found in medications, like antibiotics, including tetracycline and other pharmaceuticals. The common practice of using doses of tetracycline to cure facial, acne blemishes has been known to contribute to the discoloration of teeth. Aside from these chemicals, even excess brushing has been can create discoloration by repeated contact with fluoride compounds commonly present in most toothpastes.

Brief Summary Text (7):

Many have attempted to confront the problem of tooth discoloration by proposing various solutions to whiten the teeth. One such method of tooth whitening involves the bonding of veneers onto the buccal or labial surfaces of a tooth. The veneer is usually constructed and applied by a dentist using dental bonding techniques to attach it to the tooth. Various veneers have been described in several U.S. Pat. Nos., see, e.g. 4,992,049 "Method for Applying a Veneer Facing to a Tooth"; 4,822,279 "Article for Cosmetic Restoration of Anterior Teeth" (which uses a glazed porcelain labial veneer); 4,682,950 "Device and Method of Bonding and Veneering Dental Material to a Tooth" (synergizing composite material to a tooth surface which has been etched and coated with a bonding material); 4,473,353 "Method for Cosmetic Restoration of Anterior Teeth" (wherein a glazed porcelain veneer is bonded to a patient's tooth); and 4,433,959 "Composite Laminate Dental Veneer Containing Color

Systems" (a veneer which is molded and then attached to the labial enamel surfaces of teeth).

Brief Summary Text (8):

Other attempts to whiten teeth are also known to include bleaching the teeth. Often the bleach is applied in the form of hydrogen peroxide, which can be obtained in drug stores by consumers. Because of the delicacy of applying hydrogen peroxide in one's mouth, some dentists carry out the procedure in their offices, using a stronger peroxide than can be purchased by the consumer. There are even pastes, sold over the counter, to the consumers which claim to whiten teeth. Often ordinary toothpastes make this claim, but increasingly appearing in the marketplace for purchase by consumers are pastes with the chemical compound sodium bicarbonate (baking soda), which may also contain peroxide. While chemical bleaching of teeth has been done to provide whiter-looking teeth, with its use there exists danger to the enamel of the teeth, especially if excessive exposure to chemical bleaches occurs. Further, chemical bleaching is understood in many cases to require multiple applications, and, hence, repeated use of the chemical. Even when applied by a dentist, precautions may be taken to prevent peroxide solution from contacting the patient's gums, which if otherwise allowed to come into contact therewith can be painful and cause damage to the gums. In cases of certain stains, bleaching may not be effective, and the stain may remain.

Brief Summary Text (9):

There are even procedures involving abrasion of the tooth enamel to present a smooth surface which is lighter in appearance than the stained surface removed. This has limitations as to the number of times it can be done.

Brief Summary Text (10):

Furthermore, abrading or bleaching teeth can have deleterious side effects, including, increased sensitivity of the treated tooth to temperature, i.e. especially when hot and cold foods and drinks are consumed. This effect may subside within time, but often the need to repeat bleaching procedures regularly, gives rise to a period of time within which the treated tooth can be hypersensitive.

Brief Summary Text (11):

There are some prior art whitening methods which require etching steps that are carried out with phosphoric acids. The use of phosphoric acid is generally done by a dentist under controlled conditions, for example, in the dentist's office. Caustic acid etchants have been recognized to be corrosive to the soft tissues of the mouth. For example, ortho-phosphoric acid, in some venues, must be transported pursuant to specified requirements and restrictions. In addition, the long term physiological effects of acid etching, which are generally unknown, have led practitioners to question certain acid etching uses in the field of dentistry. See e.g. M. G. Buonocore, "The Challenge of Bonding to Dentin", The Acid Etch Technique, L. M. Silverstone and I. L. Dogon, Eds., Proceedings of the International Symposium at St. Moritz, Switzerland, Dec. 16-18, 1974, North Central Publishing Co. (St. Paul, 1975). See also, U.S. Pat. No. 5,304,585, which raises these concerns, the complete disclosure of which is herein incorporated by reference; and see Akira Komori, and Haruo Ishikawa, "Evaluation of a Resin-Reinforced Glass Ionomer Cement for Use as an Orthodontic Bonding Agent," The Angle Orthodontist, Vol. 67 No. 3, 1997, the complete disclosure of which is herein incorporated by reference. Further attempts to whiten teeth are disclosed in U.S. Pat. Nos. 4,032,627 "Tooth Whitening Cosmetic Composition"; 4,097,994 "Dental Restorative Composition Containing Oligomeric Bis-GMA Resin and Michler's Ketone"; 4,141,144 "Dental Material and Method For Controlling Tooth Lustre"; and 4,512,743 "Method for Masking Discoloration on Teeth." U.S. Pat. Nos. 4,512,743 and 4,141,144, each use phosphoric acid application to the tooth in their treatments. U.S. Pat. No. 4,097,994 discloses a photocurable compound, which is used with a specific ultraviolet sensitizer and a peroxide catalyst to cure the compound. Furthermore, phosphoric acid etching generally disposes grooves in the tooth enamel in the nature of about 50 to 60 .mu.m. This order of etching is visible and is noticed in the form of a dull tooth surface.

Brief Summary Text (12):

U.S. Pat. No. 4,032,627, referenced above, discloses the use of an alcohol-soluble composition to be applied to the surface of a tooth to whiten the tooth's

appearance. This composition is suggested to be applied by the user. However, although this disclosure attempts to provide an extended wearing time for its compound, the composition is readily worn off by the abrasive action of food eaten after the compound is applied to the teeth, with certain, more abrasive, harder, foods causing faster wear of the composition from the tooth than other, softer, foods.

Brief Summary Text (13):

A need exists for a temporary tooth whitening and colorizing system which can be safely applied by the wearer, or without the need for specially trained dental personnel. The tooth whitening to be achieved should also have stability and be able to resist removal or solvation by foods and beverages, but should be able to be removed whenever the user desires. It is further desirable to provide a composition and a method for covering a tooth, to appear white or colorized, which can be selectively applied and removed by a wearer without harming the tooth.

Brief Summary Text (15):

A novel composition and method for altering the appearance of teeth is provided by the present invention, where an individual, in his or her own home, or in a beauty parlor, or other non-medical office, can apply a colorized compound to his or her teeth to cosmetically alter and/or improve the appearance of the teeth. The present method can be used to whiten anterior teeth, and can even be done in the presence of existing saliva which may be present on the tooth. The method provides a removable coating which can comprise a colorized coating which can be removed and replaced with other colorized coatings, or no further coatings, at the user's discretion. Expensive or dangerous drying apparatus are not required. Further, the present invention can be applied with or without an etching step, thereby avoiding the hazards inherent to use of caustic acid etchants, such as phosphoric and phosphonic acids. Moreover, when an individual's teeth, in accordance with the present method, require pre-treatment prior to application of the covering compound, the application of a naturally occurring substance can be used to facilitate adhesion. The substance can be lemon juice or lime juice, which unlike caustic acids, contains citric acid, which can be purchased by the consumer user of the covering compound which is to be applied by the present method. In a proposed alternate embodiment of the invention, polyacrylic acid is applied to the tooth surface prior to contact with the covering compound.

Brief Summary Text (16):

The present invention also provides a novel method for temporarily changing the color of a tooth. A palette system is provided wherein a user or wearer can select a color from one, or a combination of one or more, colorizing compounds. The selected or created color can then be incorporated with the other steps of the present method to provide a unique appearance to a tooth. The present method permits the user to mix his or her own colors or to select a color from one or more prepared provided colors.

Brief Summary Text (17):

The composition is a removable compound which can be applied to a tooth surface. Preferably, the composition includes a lac material, which is provided with a colorizing regulating compound or substance, in a solvent such as ethanol. Lac, the natural resinous substance excreted by an insect, Laccifer Lacca, has been used in dentistry mainly for the construction of special trays and bases for wax rims when recording jaw relationships. (See A. Azucca, R. Huggett, and A. Harrison, "The Production of Shellac and its General and Dental Uses: A review." Journal of Oral Rehabilitation, 1993, vol. 20, pp. 393-400, the complete disclosure of which is herein incorporated by reference; and I. Klineberg and R. Earnshaw, "Physical Properties of Shellac Baseplate Materials." Australian Dental Journal, October, 1967, vol. 12 no.5, pp. 468-475.) Another use of shellac in dentistry includes treatment of a cavity with a hydrophilic shellac film placement of a polystyrene liner. (See M. Blixt and P. Coli, "The Influence of Lining Techniques on the Marginal Seals of Class II Composite Resin Restorations" Quintessence International, vol. 24, no.3, 1993). Shellac has also been prepared and used in dentistry for the use of a bead adhesive for securing a composite resin veneer cast restoration. (See C. Lee, H. Pierpont, and E. Strickler, "The Effect of Bead Attachment Systems on Casting Patterns and Resultant Tensile Bond Strength of Composite Resin Veneer Cast

Restorations", The Journal of Prosthetic Dentistry, November, 1991, vol. 66, no.5, pp. 623-630.)

Brief Summary Text (18):

It is an object of the present invention to provide a novel composition and method for improving the appearance of teeth.

Brief Summary Text (19):

It is a further object of the present invention to provide a composition and a method that can be useful for whitening the teeth to improve or alter their appearance.

Brief Summary Text (20):

It is a further object of the present invention to improve or alter the appearance of teeth by covering stains or discolorations on a tooth by matching a colored compound to an individual's teeth and applying the colored compound on the surface of said stained or discolored teeth.

Brief Summary Text (21):

A further object of the present method is to improve the appearance of a tooth by applying a colored or shaded composition to the tooth.

Brief Summary Text (22):

A further object of the present invention is to coordinate colors of a tooth with other health and beauty aids by applying a method for colorizing a compound and applying it to a tooth.

Brief Summary Text (23):

Another object of the present invention is to provide a method for applying a compound to a tooth which can remain on the tooth during eating and other activity, and can be removed at the wearer's discretion, to leave the tooth as it appeared before the application of the compound.

Brief Summary Text (24):

A further object of the present invention is to provide a novel method of improving the appearance of a tooth which includes applying a compound to the tooth and selectively removing the compound from the tooth.

Brief Summary Text (25):

A further object of the present invention is to provide a method for improving the appearance of a tooth which can be repeated by the wearer, to change the appearance of a tooth, on a regular basis if the wearer desires, without having damaging effects on the tooth enamel.

Brief Summary Text (26):

Another object of the present invention is to provide a method which includes a color matching system wherein the user mixes components to colorize the composition to be applied to the tooth to the desired hue or color, which can be a tooth color or a non-tooth color.

Brief Summary Text (27):

It is another object of the present invention to accomplish the above objects by repeating application and removal of the compound on a tooth for different colors as the user determines and selects.

Brief Summary Text (28):

Another object of the present invention is to provide a tooth covering composition which can be selectively colorized, applied and removed by a user.

Brief Summary Text (29):

Another object of the present invention is to provide a compound which can be selectively removed from the tooth by a forced stream of water.

Brief Summary Text (31):

The present invention provides a method for altering the appearance of teeth. The

altered appearance can be the removal of stains or discoloration caused by disease, chemical exposure or aging, or can be the application of a color (i.e. red, blue green, etc) to the tooth. Generally the enamel of teeth is bound to tannins and other chemicals which remain on the enamel, in the form of a stain, which cannot be removed by brushing.

Brief Summary Text (32):

The present invention provides a composition which can be selectively applied to a tooth surface to provide a cosmetic alteration of the tooth, specifically, whitening or colorizing the tooth. The compound, which is a lac based compound, preferably can be provided, or can be mixed by the user, to colorize the tooth a tooth colored shade, or one or more of a variety of colors (i.e. red, blue, green etc.). The present method provides for the application of a lac based compound to the surface of a tooth to cover discolorings which are present on the tooth, or to provide a colorization of the tooth.

Brief Summary Text (33):

A pigment compound is used to regulate the color of the tooth which is to be covered with the covering compound. For example, one pigment which can be used is titanium dioxide, which can yield a white or tooth colored shade. Other suitable pigments can be used, either separately, or combined to form desired shades.

Brief Summary Text (34):

The covering compound to be applied by the present method comprises an opaque material which masks the tooth surface. Preferably, the covering compound comprises a lac based compound.

Brief Summary Text (35):

The compound is prepared by dissolving a lac material into a solvent. The solvent used for the example below is ethanol (99% ethyl alcohol). The compound is preferably a liquid form which is fluid and can be painted on the surface of a tooth.

Brief Summary Text (41):

The covering compound is applied to the teeth in the present invention by painting it onto the tooth surface, preferably with a brush. This enables the composition to be evenly distributed to cover the entire tooth, and furthermore, without an excessive buildup or absence of material in one spot of the tooth. Alternately an aerosol suspension can be used to apply the covering compound to the tooth. For example, a small tube can be used through which the aerosol can be propelled for application onto the tooth. This facilitates even distribution of the covering compound on the tooth surface and avoidance of coverage on gums, skin and other non-tooth surfaces.

Brief Summary Text (42):

The present method also provides palette means for controlling the colorizing of the tooth. The palette means preferably comprises a plurality of pigments which are present in individual quantities for adding to the covering composition to be applied to the tooth. The palette means preferably is provided with a plurality of pigment means which contain tooth-colored pigments which can be matched to the individual user's tooth color. In addition, the palette means can contain colorful shades, such as, for example, blue, pink, pastel colors, or any other color which is supplied in the form of a pigment compound which can be mixed with the base covering compound and then be applied to the tooth by painting on the tooth.

Brief Summary Text (43):

The method can be carried out, for example, by providing a tray having a series of compartments containing tooth-colored pigments arranged in one row and a series of colorizing pigments in other rows. A larger compartment can be provided for the base compound or components. The compartments can have resealable covers which can be opened and closed by the user to remove a pigment or compound as needed. A mixing area can also be provided and can also have a cover.

Brief Summary Text (44):

Alternately, it is possible to apply the lac-based covering compound on the tooth,

and then mix the color or pigment compound with the lac compound directly on the tooth. This may be done, for example, to facilitate approximating the shade of adjacent teeth.

Brief Summary Text (45):

In some circumstances, it may be desirable to etch a tooth surface to provide microscopic pits for facilitating adhesion of a compound to be applied thereto. Etching provides an increased surface area of the tooth. However, the present method also requires removability of the temporary tooth coating. In a preferred embodiment of the invention, the teeth of an individual can be pretreated with citric acid, of the same weakness of that concentration contained in lemon juice. This facilitates the adherence of the covering compound on the tooth's surface. The citric acid prepares the surface of the tooth to receive the compound which is to be painted on the tooth.

Brief Summary Text (46):

The method contemplates application of the tooth enhancing composition by an individual user or wearer having no special training or knowledge in dentistry. In this case, lemon juice can be used, since it will be easily obtained by the user. Alternately, citric acid solutions can be prepared or provided having the same general concentration as citric acid in lemon juice. By the use of the etching step, very small grooves or pits are formed on the tooth surface, which are approximately 5 to 10 .mu.m or less. This enables the wearer of the compound to restore a treated tooth to its original appearance when desired by removing the covering compound. Another acid proposed for use with the compound of the present invention, for tooth preparation is polyacrylic acid.

Brief Summary Text (47):

Alternately, it is proposed that the covering compound, for example, can be prepared by taking a lac based composition, such as of the type described in Examples 1 or 2, comprising a 1 g. lac/10 ml ethanol solution, or in Examples 3 or 4, and providing an amount of a pigment compound which becomes the base pigment. To this pigmented base compound, the user can customize the color with the colorizing means, by selecting and combining pigment items and admixing the selected pigment items with the base compound. In addition, other elements, such as reflective means can be admixed as well to provide additional unique alteration of the tooth. For example, polymer particles, such as glitter, can be added to provide a sparkle effect to the tooth.

Brief Summary Text (48):

A colorizing pigment can be supplied in the form of a modified food starch or other color additive. The pigment can be supplied with the lac compound, or can be provided separately to be admixed by the user with the lac compound. The lac compound can be supplied to the user with a pigment to exhibit a tooth-colored shade, and can even be further mixed for colorization by the user.

Brief Summary Text (50):

The form of the additive for use in the present invention preferably includes dye form additives, but may also include lake forms which are compatible with the lac based covering composition. Water soluble dyes, provided in the form of powders, granules, liquids or other special-purpose forms can be used in accordance with the present method. Lakes, the water insoluble form of the dye, are generally used for coloring products which do not contain adequate moisture to dissolve the dyes. For example, if a suspension of color is to be used, a lake form additive can be employed. The color additive provided in the form of a lake may, for example, be used with other tooth appearance enhancing means such as glitter particles.

Brief Summary Text (51):

The present method includes the step of painting the compound on a tooth which is in its in vivo environment and an intermediary is not required. The naturally occurring saliva may be present on the tooth, and the compound can be applied with the saliva being present. Alternately, the method can include applying the compound to a tooth by spraying. For example, a compressed gas propellant, such as an aerosol, can be utilized to provide delivery of the covering compound to the tooth.

Brief Summary Text (52):

The selected compound is painted on the tooth and permitted to harden. The hardening of the compound occurs within about two to three minutes, wherein the alcohol dries or evaporates and the solubilized lac composition polymerizes. A layer is then formed by the compound covering the tooth surface on which it was applied, and becomes fixed on the tooth. The tooth thereby exhibits a new appearance, attributable to the compound.

Brief Summary Text (53):

The compound exhibits permanence and withstands normal buccal functions such as, for example, brushing, eating, chewing, contacting foods and beverages, and other functions carried out with one's teeth. The method applies a covering on the tooth which maintains a uniform appearance and is further resistant to staining. In addition, the covering protects the tooth against further staining and contact with bacteria and chemicals. The method applies a thin coating to the tooth which does not interrupt the user's normal mouth functions, and does not feel uncomfortable to a user.

Brief Summary Text (54):

The method further includes selectively removing the coating. The coating is removably provided on the tooth and can be removed from the tooth at the user's discretion. For example, if a user desires to change the color of the coated tooth, and, for example, apply a different color, then the first coating is removed to expose the original tooth surfaces again. A second or next coating can then be applied to the tooth to change the color. Removing preferably includes the step of using a pressurized stream of water, which can be done with a commercially available appliance, such as, for example, a WATER PIK.RTM..

Brief Summary Text (55):

Alternately, it is proposed that a solvent may be used to remove the tooth coating. Preferably, the solvent comprises a composition which the user can readily obtain, or which can be supplied to the individual, non-medical personnel user. Furthermore, it is conceivable that compatible solvents can be used for removing the covering compound from a tooth by softening or dissolving the compound or its bonds. The solvent, for example, can be a material which may break up bonds between the lac polymerization and/or invade a bond formed between the compound and the tooth surface. The removal step wherein a solvent is used, can be accomplished by swabbing the solvent onto the tooth coating or around the edges thereof with a cotton swab. The coating is then loosened and can be more easily removed, or can even be dissolved.

Brief Summary Text (56):

Removal of the covering coating from the tooth then displays the original surface of the underlying tooth which had been covered by the coating.

Brief Summary Text (57):

The following is an example of the use of the lac based covering compound and the method of the present invention, as carried out on human teeth in vivo. The dewaxed orange flake lac resin was used as indicated in Examples 1 and 2, below, to formulate a covering compound for cosmetically altering the appearance of a tooth, although it is conceivable that other lac resins, such as white lac, can also be used consistent with the scope of the present invention. While ethanol is described as a preferred solvent, it will be understood that an ethanol solution of less than 99% pure ethanol may be utilized, however, the drying times may be affected by the addition of alternate solvents.

Detailed Description Text (2):

An anterior tooth was prepared by retracting the individual's lip to expose the entire tooth, up to and including the upper gum line. The tooth was a front upper tooth. A quantity of 1 g. of lac resin (obtained commercially from A.F. Suter & Co. Ltd. as "Dewaxed Orange Flake Shellac" as discussed above), a dry light brown flake material was dissolved in 10 ml of grain alcohol (99% ethanol). A coloring agent was then added to tint the lac mixture to the desired shade. In this example, titanium dioxide powder (obtained commercially from Gamblin Dry Pigments, P.O. Box 625, Portland, Oreg. 97207) was admixed with the lac solution. The amount of titanium

dioxide powder brought the color of the amber solution to a tooth colored shade, and the addition was an amount sufficient to arrive at the desired shade.

Detailed Description Text (3):

The covering compound was then applied onto the surface of the tooth. The covering compound dried within two minutes of its application by exposure to the air, the alcohol evaporating from the solution leaving a lac coating on the tooth. The result was an evenly-coated tooth, which presented an evenly-colored appearance. Any discoloration or uneven shading which was previously present on the tooth was no longer visible.

Detailed Description Text (4):

The tooth was used normally, for eating, drinking, and was brushed regularly for a four-day period, after which the coating on the tooth was easily removed by the application of a high pressure stream of water (supplied by the use of a WATER PIK.RTM., an instrument which applies a pressurized stream of water onto the tooth, and which is commercially available to consumers). The coating was abraded and removed from the tooth with a WATER PIK.RTM..

Detailed Description Text (6):

The above conditions were repeated, as reported for Example 1, above, for an upper front tooth. However, the tooth was first prepared by exposing the tooth to citric acid (applied in the form of lemon juice). The lemon juice was permitted to remain on the tooth for one minute, after which time, it was washed off of the tooth with a water rinse. The lac based covering compound was then prepared and applied to the washed tooth, which was still wet, in accordance with the same procedure as in Example 1, above. This coating performed, and could be removed, in the same manner as the coating in Example 1, above.

Detailed Description Text (8):

An upper front tooth was prepared by retracting the individual's lip to expose the entire tooth, up to and including the upper gum line. The tooth was a front upper tooth. A quantity of about 0.10 mls of a lac resin solution (containing bleached food grade (USP) dewaxed lac resin solubilized in specially denatured alcohol formula 45/200 proof (SDA 45/200), obtained commercially from Mantrose-Hauser Company, 1175 Post Road East, Westport, Conn. 06880, USA) was taken on a sable brush. The solution was a clear color.

Detailed Description Text (9):

A coloring agent was then added to tint the lac solution to a desired shade. In this example, titanium dioxide powder (obtained commercially from Gamblin Dry Pigments, P.O. Box 625, Portland, Oreg. 97207) was admixed with the sample of lac solution taken on the brush. The sable brush deposited the lac solution sample onto a clean dish. An amount of pigment, equal to the size of a pinhead was added to the dish containing the lac solution sample were admixed using the brush, until a uniformly pigmented covering compound result (i.e. wherein the pigment was uniformly distributed throughout the compound). The amount of titanium dioxide powder brought the color of the clear solution to a tooth colored shade, and the addition was an amount sufficient to arrive at the desired shade. The covering compound was brushed onto the tooth surface. The covering compound dried within minutes of its application. The tooth was used normally, for eating, drinking, and was brushed regularly for a four-day period, after which the coating on the tooth was easily removed by the application of a high pressure stream of water (supplied by the use of an instrument which applies a pressurized stream of water onto the tooth, and which is commercially available to consumers, such as, for example; a WATER PIK.RTM.). The coating was abraded and removed from the tooth with the WATER PIK.RTM..

Detailed Description Text (11):

The above conditions were repeated, as reported for Example 3, above, for a front upper tooth. However, the tooth was first prepared by exposing the tooth to citric acid (applied in the form of lemon juice). The lemon juice was permitted to remain on the tooth for one minute, after which time, it was washed off of the tooth with a water rinse. The lac based covering compound was then prepared and applied to the washed tooth, which was still wet, in accordance with the same procedure as in

Example 3, above. This coating performed, and could be removed, in the same manner as the coating in Example 3, above.

Detailed Description Text (13):

The above conditions were repeated, as reported for Example 3, above, for an upper front tooth. The covering compound was prepared by taking a quantity of about 0.10 mls of a lac resin solution containing bleached food grade (USP) dewaxed lac resin solubilized in specially denatured alcohol formula 45/200 proof (SDA) 45/200, (obtained commercially from Mantrose-Hauser Company, 1175 Post Road East, Westport, Conn. 06880, USA) which was taken on a brush and mixed with a quantity of pigment in a dish. The preparation of the covering compound was in accordance with the procedure identified above in Example 3, however, instead of titanium dioxide powder, a red pigment (red # 22, sold commercially and obtained from Warner Jenkinson Co. Inc., 107 Wade Avenue, South Plainfield, N.J. 07080 as K-7008) was used. The compound was mixed and a red color resulted. The red coloring compound was applied to a tooth in the manner described in connection with Example 3. The tooth, having the red color from the compound applied to it, was used normally, for eating, drinking and was brushed regularly for a four-day period, after which the coating on the tooth was easily removed by the application of a high pressure stream of water (supplied by the use of an instrument which applies a pressurized stream of water onto the tooth and which is commercially available to consumers, such as, for example, a WATER PIK.RTM.). However, the tooth was first prepared by exposing the tooth to citric acid (applied in the form of lemon juice). The lemon juice was permitted to remain on the tooth for one minute, after which time, it was washed off of the tooth with a water rinse. The lac based covering compound was then prepared and applied to the washed tooth, which was still wet, in accordance with the same procedure as in Example 3, above. This coating performed, and could be removed, in the same manner as the coating in Example 3, above.

Detailed Description Text (14):

This example was also repeated using, in place of the red pigment, yellow # 6 powder (FD&C 08006), and again using D & C yellow #10 (K-7059) (Warner-Jenkinson). Similar results were obtained, however instead of a red color on the tooth, a yellow or orange color was obtained.

Detailed Description Text (17):

The covering compound is initially prepared, in accordance with Example 1, or by starting with the lac solution of Example 3. The covering compound is then further prepared by admixing with it a colorizing pigment. The colorizing pigment can be selected from color additives, including titanium dioxide and other pigments, vegetable dyes and the like. In this manner a variety of tooth-colored shades are possible, and can be matched to existing or surrounding teeth shades, by approximation with the addition of a pigment.

Detailed Description Text (18):

In this example, it is proposed that the covering compound is provided in a tooth colored shade to the user. The provided covering compound is then admixed with a selected pigment to provide a colorized compound. The colorized compound is applied to a tooth surface in the manner recited above in Examples 1 or 3, by painting it on the tooth surface. The compound is then permitted to dry by exposure to air, and selectively removed at the wearer's discretion.

Detailed Description Text (21):

The lac based covering compound can be provided in a tooth colored shade which can be further customized by the user with a selection from the pigment compounds. The tooth colored shade can preferably be provided by the addition of titanium dioxide or other suitable pigment, or by varying the types of lac used, or both.

Detailed Description Text (22):

The colorized covering compound is applied to a tooth surface in the manner recited above in Examples 1 or 3. The covering compound is then permitted to dry by exposure to air, and selectively removed at the wearer's discretion.

Detailed Description Text (24):

The compounds and methods described in Examples 1, 3, 5, 6 and 7, above, but further

including an etching step. A weak acid is provided to etch very small microscopic pits onto the tooth surface. A citric acid solution is used. The citric acid solution preferably has the same concentration as lemon juice and, further, can be used in the form of lemon juice. The lemon juice is applied onto the tooth surface which is to receive the covering compound and allowed to remain on the tooth from about a few seconds to a few minutes. The lemon juice is then washed from the tooth with a water rinse. The tooth, still wet, is now ready to receive the covering compound. The covering compound is then applied by brushing onto the tooth surface to provide an evenly dispersed coating on the tooth. The compound is then permitted to dry by exposure to air, and selectively removed at the wearer's discretion.

Detailed Description Text (26):

The method is carried out as in Example 8, wherein the tooth preparing step includes etching the tooth with a polyacrylic acid solution (in place of the citric acid solution) by contacting the tooth with the polyacrylic acid solution and allowing the acid solution to remain on the tooth for a couple of minutes. The polyacrylic acid solution is then rinsed off of the tooth by applying a water rinse. Thereafter, the covering compound is applied.

Detailed Description Text (28):

The method is carried out as in any of Examples 1-9 above, wherein the covering compound is provided having a powdered component and a liquid component. The powdered component can contain the lac base and a pigment, which can be a tooth colored pigment or a non-tooth colored pigment. The powdered component can be provided in a plurality of pigmented shades for selection by the user. The powdered component is selected and mixed with a liquid component, which, for example, can contain ethanol, to form the lac based covering compound for application to an etched or non-etched tooth.

Detailed Description Text (31):

In each of the above examples, it is noted that no effort is made to dry the tooth, prior to applying the covering compound thereon.

Other Reference Publication (1):

Klineberg I. And Earnshaw R., "Physical Properties of Shellac Baseplate Materials." Australian Dental Journal. Oct., 1967, vol. 12, No. 5, pp. 468-475.

Other Reference Publication (2):

Azouka A., Hugget R., and Harrison A. "The Production of Shellac and its General and Dental Uses: A Review." Journal of Oral Rehabilitation. 1993, vol. 20, pp. 393-400.

Other Reference Publication (8):

Silverman E., Cohen M., Demke, and Silverman M., "A New Light-Cured Glass Ionomer Cement That Bond Brackets to Teeth Without Etching in the Presence of Saliva", (American Association of Orthodon.), 1995, vol. 108, pp. 231-236.

CLAIMS:

1. A method for cosmetically altering the appearance of teeth to whiten or colorize the teeth, comprising the steps of:

- a) preparing the tooth by exposing the tooth so that the tooth surface to be altered is visible to the gum line;
- b) preparing a lac based covering compound to be applied onto the enamel surface of the tooth to be altered, including selecting a compound from a plurality of tooth colored compounds to match the shade of the adjacent or surrounding teeth in the individual's mouth;
- c) applying the covering compound prepared in step b. to the tooth surface to be coated; and
- d) allowing the covering compound applied to the tooth to dry by exposing the tooth to air.

2. The method of claim 1, wherein the step of preparing a lac based covering compound includes selecting a color from one or more non-tooth colored compounds and mixing said non-tooth-colored compound with said lac based covering compound to provide an overall non-tooth colored appearance, and wherein the covering compound is applied to the tooth surface evenly to cover the tooth surface and is permitted to cure by drying the compound to provide a non-tooth colored coating on the surface of the tooth.
3. The method of claim 2, further including the step of providing palette means, said palette means including a plurality of pigmented compounds, and selecting one or more of said pigmented compounds from said palette means and mixing said selected one or more pigmented compounds with said the lac based covering compound of step b) before applying said lac based covering compound to said tooth.
5. The method of claim 1, further including the step of selectively removing the lac based covering compound applied to the tooth.
6. The method of claim 5, wherein the step of selectively removing the lac based covering compound from the tooth includes contacting the compound with a solvent.
7. The method of claim 5, wherein the step of selectively removing the lac based covering compound includes applying to the tooth surface containing the lac based covering compound a forced stream of water.
8. The method of claim 1, further comprising the step of providing colorizing means containing a plurality of pigments to form palette means, and wherein the method further includes the steps of selecting at least one pigment from said palette means, and mixing said selected pigment with said lac based covering compound to be applied to the tooth.
9. The method of claim 1, further including the step of mixing a reflecting material into said lac based covering compound to be applied to said tooth.
12. The method of claim 1, wherein the step of preparing a tooth further includes the steps of providing a weak acid solution and contacting the tooth surface with the weak acid solution.
13. The method of claim 12, wherein the step of contacting the tooth with a weak acid solution, includes etching the tooth with an acid selected from the group of citric acid and polyacrylic acid.
14. The method of claim 1, wherein the step of preparing a tooth further includes the step of etching microscopic pits which are less than about 5.0 .mu.m in the tooth surface with an etchant.
15. The method of claim 1, wherein the step of preparing a lac based covering compound further includes selecting a food grade dye and blending said dye with said covering compound to provide a colored covering on said tooth.
16. The method of claim 1, wherein the step of preparing a lac based covering compound further comprises providing said lac based covering compound in a fluidic suspension for delivery by spraying, and wherein the step of applying the covering compound to the tooth surface comprises the step of spraying the covering compound on the tooth surface.
17. A method for cosmetically altering the appearance of teeth to whiten or colorize the teeth, comprising the steps of:
- a) preparing the tooth by exposing the tooth so that the tooth surface to be altered is visible to the gum line;
- b) providing palette means containing a plurality of tooth-colored shaded pigment compounds, including the step of providing a container means for holding the pigment compounds and providing a mixing area;

- c) selecting from said palette means one or more of said pigments contained in the container means to match the shade of adjacent or surrounding teeth in an individual's mouth;
- d) preparing a lac based covering compound to be applied onto the enamel surface of the tooth to be altered by placing said lac based covering compound in said mixing area of said container means and adding to said lac based covering compound from said container means a pigment compound from said palette means;
- e) mixing said covering compound with said pigment compound;
- f) applying the lac based covering compound prepared in step e) to the tooth surface to be coated; and
- g) drying the covering compound applied to the tooth in step f) by exposing the covered tooth to air.

18. The method of claim 17, wherein the step of preparing a tooth further comprises the steps of:

- a) providing a weak acid solution;
- b) etching the tooth by contacting the tooth with a weak acidic solution to provide small microscopic pits in the enamel surface of said tooth; and
- c) rinsing the weak acid solution off of the tooth with water.

19. A system for cosmetically altering the appearance of teeth wherein a removable coating is applied directly on a dry or wet tooth and cured on the tooth surface, comprising a lac based covering compound having a lac compound and a solvent, one or more colorizing pigment compounds, means for storing the lac based covering compound and associated means for storing one or more colorizing pigment compounds, wherein the lac based covering compound further comprises a compound selected from the group consisting of Bis-GMA sealants and glass ionomeric cements, wherein the system provides the user with the ability to select from the associated means one or more colorizing pigment compounds and mix said selected one or more colorizing pigment compounds with the lac based covering compound and to form a pigmented covering compound, apply the pigmented covering compound to cover the surface of the tooth to be altered, permit the pigmented covering compound to dry by exposure to air, and remove the pigmented covering compound from the tooth at the user's discretion.

20. A composition for cosmetically altering the appearance of teeth to whiten or colorize the teeth by applying said composition to a tooth surface, said composition comprising:

- a) a lac resin;
- b) a solvent for dissolving said lac resin; and
- c) a pigment;
- d) wherein the concentration of said lac resin is approximately 1 g/10 ml solvent, and wherein the lac resin is present in the composition in an amount which is greater than the amount of pigment in the composition.

25. The composition of claim 20, wherein said pigment comprises titanium dioxide.

28. A covering composition for application to the surface of a tooth for cosmetically altering the appearance of the tooth to whiten or colorize the tooth, said covering composition comprising dewaxed orange flake lac resin solubilized in an ethanol solution at a concentration of 1 g. per 10 ml of ethanol, and a pigment.

30. The covering compound of claim 28, wherein said pigment comprises titanium dioxide.

31. The covering compound of claim 28, wherein said pigment comprises a non-tooth colored shade for cosmetically colorizing the tooth when the covering compound is applied thereon.

32. A method for cosmetically altering the appearance of teeth to whiten or colorize the teeth, comprising the steps of:

- a) preparing the tooth by exposing the tooth so that the tooth surface to be altered is visible to the gum line;
- b) preparing a lac based covering compound to be applied onto the enamel surface of the tooth to be altered, including selecting a compound from a plurality of tooth colored compounds to match the shade of the adjacent or surrounding teeth in the individual's mouth;
- c) applying the covering compound prepared in step b) to the tooth surface to be coated;
- d) allowing the covering compound applied to the tooth to dry by exposing the tooth to air;
- e) wherein the step of preparing a tooth further includes the steps of providing a weak acid solution and contacting the tooth surface with the weak acid solution; and
- f) wherein the weak acid solution is a citric acid of lemon juice.

33. A composition for cosmetically altering the appearance of teeth to whiten or colorize the teeth by applying said composition to a tooth surface, said composition comprising:

- a) a lac resin;
- b) a solvent for dissolving said lac resin;
- c) a pigment;
- d) wherein said solvent comprises ethanol; and
- e) wherein said lac resin comprises refined bleached food grade dewaxed lac.

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L4: Entry 21 of 37

File: USPT

Jul 7, 1998

US-PAT-NO: 5776437

DOCUMENT-IDENTIFIER: US 5776437 A

TITLE: Method of making a tartar control dentifrice containing fluoride and peroxide

DATE-ISSUED: July 7, 1998

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
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Britt; Michael Lashawn	Cincinnati	OH		

US-CL-CURRENT: 424/53; 424/49, 424/52, 424/57

CLAIMS:

What is claimed is:

1. A method of manufacturing predominately undissolved pyrophosphate and calcium peroxide dentifrice compositions comprising the steps of:

a. preparing a mixture of a soluble fluoride ion source and one or more aqueous carrier materials;

b. adding tetrasodium pyrophosphate and calcium peroxide, all at once or in portions, under conditions wherein less than about 20% of the total pyrophosphate and calcium peroxide are dissolved in the mixture, and wherein any further remaining aqueous carrier materials not added to the mixture during step (a) are added in whole or in part in step (b) or thereafter, either by themselves or with any remaining amount of the tetrasodium pyrophosphate or calcium peroxide under conditions such that less than about 20% of the total pyrophosphate and calcium peroxide are dissolved in the mixture; and

c. heating the mixture to a temperature range of from about 38.degree. C. to about 71.degree. C.;

and wherein the dentifrice has a viscosity of from about 10 to about 60 Brookfield units at 23.degree. C. in bulk or packed product from about 10 minutes to about two hours after being made or packed and the viscosity builds to about 30 to about 125 Brookfield units at 23.degree. C. in bulk or packed product after about one month or more after being made or packed.

2. The method of manufacturing a composition according to claim 1 wherein the tetrasodium pyrophosphate and calcium peroxide are added to the mixture after all other sodium-containing salts present in the composition have been added to the mixture.

3. The method of manufacturing a composition according to claim 2 wherein the mixture has a neat pH of above about pH 8 during and after the tetrasodium pyrophosphate and calcium peroxide additions are made to the mixture.

4. The method of manufacturing a composition according to claim 1 (c) further comprising controlling one or more heating variables selected from the group consisting of rate of heating the mixture to a temperature range of from about 38.degree. C. to about 71.degree. C., amount of time mixture is kept at the temperature range, rate of cooling the mixture from the temperature range, homogenization of the mixture while the mixture is within the temperature range, and combinations thereof.
5. The method of manufacturing a composition according to claim 4 wherein the tetrasodium pyrophosphate is in an amount of at least about 1.5%.
6. The method of manufacturing a composition according to claim 5 wherein the calcium peroxide is in an amount of from about 0.01% to about 5%.
7. The method of manufacturing a composition according to claim 6 wherein the soluble fluoride ion source is sodium fluoride and is capable of providing from about 50 ppm to about 3500 ppm of free fluoride ions.
8. The method of manufacturing a composition according to claim 7 wherein the composition has a neat pH of from about 9.0 to about 10.5.
9. The method of manufacturing a composition according to claim 8 wherein the composition has a total water content of from about 5% to about 20%.
10. The method of manufacturing a composition according to claim 9 wherein from about 0.5% to about 40% of an alkali metal bicarbonate salt is added in step 1 (a).
11. The method of manufacturing a composition according to claim 10 wherein from about from about 0.01% to about 25% of xylitol is added in step 1 (a).
12. The method of manufacturing a composition according to claim 11 wherein from about 0.25% to about 10% of a nonionic surfactant is added in step 1 (a).
13. The method of manufacturing a composition according to claim 12 wherein the nonionic surfactant is poloxamer.
14. The method of manufacturing a composition according to claim 13 wherein from about 80% to about 98% of one or more of the aqueous carriers materials are added in step 1 (a).
15. The method of manufacturing a composition according to claim 14 wherein the aqueous carriers contain materials selected from the group consisting of thickening materials, humectants, water, buffering agents, surfactants, abrasive polishing materials, sweetening agents, flavor systems, coloring agents, titanium dioxide, and mixtures thereof.

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L4: Entry 22 of 37

File: USPT

Nov 18, 1997

US-PAT-NO: 5688492

DOCUMENT-IDENTIFIER: US 5688492 A

TITLE: Oral hygiene composition

DATE-ISSUED: November 18, 1997

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
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Cooper; Michael David	Nottingham			GB2

US-CL-CURRENT: 424/49; 424/421, 424/440, 424/464, 424/468, 424/48, 424/489, 424/52,
424/53, 424/54, 424/600, 424/604, 424/606, 424/617, 424/618, 424/630, 424/635,
424/641, 424/642, 424/643, 424/649, 424/650, 424/688, 424/691

CLAIMS:

We claim:

1. An oral composition suitable for use in oral hygiene or dental treatment comprising an effective amount of an antimicrobial agent in the form of a particulate, non-ion-exchanging, zeolite-free, inert metal oxide, a sparingly soluble metal salt providing antimicrobial metal ions in use and a further material selected from the group consisting of humectants, gelling agents, abrasives, fluoride sources, desensitizing agents, flavorings, colorings, sweeteners, preservatives, structuring agents, bactericides, anti-tartar agents and anti-plaque agents.
2. A composition as claimed in claim 1, wherein the metal salt is selected from the group consisting of silver, copper, gold, platinum and zinc salts.
3. A composition as claimed in claim 1, wherein the metal oxide is selected from the group consisting of titanium dioxide, aluminum oxide, zirconium oxide, hafnium oxide, niobium oxide, tantalum oxide, calcium hydroxyapatite, barium sulphate, and magnesium oxide.
4. A composition as claimed in claim 3, wherein the titanium dioxide is in at least one of anatase, rutile and brookite crystalline form.
5. A composition as claimed in claim 1 wherein the antimicrobial agent is employed in combination with a further antimicrobial agent providing short-term antimicrobial action.
6. A composition as claimed in claim 1 wherein the metal salt is present in an amount of from about 0.5 to 75% by weight of the antimicrobial agent.
7. A composition as claimed in claim 6 wherein the metal salt is present in an amount of from about 1 to 50% by weight of the antimicrobial agent.
8. A composition as claimed in claim 7 wherein the metal salt is present in an amount of from about 2 to 25% by weight of the antimicrobial agent.
9. A composition as claimed in claim 1 wherein the metal oxide has an average

particle size of less than about 25 micrometers.

10. A composition as claimed in claim 9 wherein the average particle size is between about 0.01 and 15 micrometers.

11. A composition as claimed in claim 1 wherein the metal oxide is titanium dioxide having a primary crystal size of from about 0.005 to 5 micrometers.

12. A composition as claimed in claim 11 wherein the titanium dioxide has a primary crystal size of from about 0.01 to 1 micrometers.

13. A composition as claimed in claim 1 wherein the metal oxide has a mean surface area of from about 1 to 300 m.² /g.

14. A composition as claimed in claim 1 wherein the antimicrobial agent is included in the composition in an amount of from about 1.times.10.⁻⁵ to 5% by weight.

15. A composition as claimed in claim 14 wherein the antimicrobial agent is included in the composition in an amount of from about 0.01% to 3% by weight.

16. A method of cleaning a mouth of an individual for cosmetic purposes, comprising applying to the mouth an oral hygiene composition comprising an effective amount of an antimicrobial agent comprising a particulate, non-ion-exchanging, zeolite-free, inert metal oxide and a sparingly soluble metal salt providing antimicrobial metal ions in use.

17. A method of treating or preventing dental caries or gingivitis, comprising applying to a mouth of an individual an oral hygiene composition comprising an effective amount of an antimicrobial agent comprising a particulate, non-ion-exchanging, zeolite-free, inert metal oxide and a sparingly soluble metal salt providing antimicrobial metal ions in use.

18. A method of providing sustained antimicrobial activity in a mouth of an individual, comprising applying to the mouth an oral hygiene composition comprising an effective amount of an antimicrobial agent comprising a particulate, non-ion-exchanging, zeolite-free, inert metal oxide and a sparingly soluble metal salt providing antimicrobial metal ions in use.

19. A composition as claimed in claim 5, wherein the further antimicrobial agent is selected from the group consisting of a zinc salt, a tin salt, cetyl pyridinium chloride, a bis-biguanide, aliphatic amines, bromochlorophene, hexachlorophene, salicylanilides, a quaternary ammonium compound and triclosan.

20. A composition as claimed in claim 1, wherein the composition is selected from the group consisting of a dentrifice, mouthwash, tooth powder, chewing gum, lozenge, mouth spray, denture cleansing formulation, tooth paint and glass ionomer cement.

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L4: Entry 22 of 37

File: USPT

Nov 18, 1997

DOCUMENT-IDENTIFIER: US 5688492 A

TITLE: Oral hygiene composition

Brief Summary Text (1):

The present invention relates to an oral hygiene composition providing sustained antimicrobial action. There is also provided inter alia the use of such a composition in the treatment or prevention of dental caries or gingivitis and in the cleansing of the mouth for cosmetic purposes.

Brief Summary Text (2):

The term `oral hygiene composition` as used herein indicates a formulation for use in any form of oral hygiene or dental treatment and includes inter alia dentifrices, mouthwashes, toothpowders, chewing gums, lozenges, mouthsprays, flosses, denture cleansing formulations, tooth paints and glass ionomer cements.

Brief Summary Text (3):

The use of antimicrobial agents in oral hygiene compositions is well-known in the art. Typically, the anti-microbial agent is an organic species such as chlorhexidine, bromochlorophene, cetyl pyridinium chloride or benzethonium chloride. These agents are known to be effective against the microorganisms associated with dental caries and gingivitis, such as the bacteria Streptococcus mutans and Streptococcus salivarius, and the fungus Candida albicans.

Brief Summary Text (6):

The present invention seeks to solve this problem by providing an oral composition comprising an antimicrobial agent with a tendency to remain in the mouth in use, thus giving sustained antimicrobial activity and enhanced protection against dental caries and gingivitis.

Brief Summary Text (8):

JP-A-02283312 (Sanpo Seijaku KK) discloses a toothbrush having brush-hairs incorporating a bactericidal metal-carrying zeolite, the metal being carried on the zeolite by ion-exchange. The toothbrush is used for brushing teeth and gums.

Brief Summary Text (10):

It has now surprisingly been found that antimicrobial agents of the type disclosed in EP-A-0251783 are particularly well-suited to use in oral hygiene compositions. The agents in question show a high degree of substantivity to the mouth, and to the teeth and gums in particular, thus providing sustained oral antimicrobial action.

Brief Summary Text (20):

The oral composition may be formulated for use in any form of interdental or periodontal treatment and may be in the form, for example, of a dentifrice, mouthwash, toothpowder, chewing gum, lozenge, mouthspray, floss, dental paint, or glass ionomer cement. Use of the antimicrobial material of the present invention in a glass ionomer cement has the advantage of providing X-ray opacity as well as antimicrobial action.

Brief Summary Text (30):

Abrasives should preferably be capable of cleaning and/or polishing the teeth without causing harm to dental enamel or dentine. They are used most commonly in dentifrices and toothpowders, but may also be used in mouthwashes etc. Suitable abrasives include the silica abrasives, such as hydrated silicas and silica gels,

particularly silica xerogels such as those available under the trade name `Syloid` from W. R. Grace and Company. Also suitable are precipitated silica materials such as those available under the trade name `Zeodent` from J. M. Huber Corporation, and diatomaceous earths such as those available under the trade name `Celite` from Johns-Manville Corporation. Alternative abrasives include alumina, insoluble metaphosphates such as insoluble sodium metaphosphate, calcium carbonate, dicalcium phosphate (in dihydrate and anhydrous forms), calcium pyrophosphate (including .beta.-phase calcium) polymethoxylates and particulate thermosetting polymerised resins such as, for example, melamine-ureas, melamine-formaldehydes, urea-formaldehydes, melamine-urea-formaldehydes, cross-linked epoxides, melamines, phenolics, highly purified celluloses such as those available under the trade name `Elcema` from Degussa AG, and cross-linked polyesters. Suitably, abrasives are included in an amount of from 0-80%, preferably 0-60% by weight of the oral hygiene composition.

Brief Summary Text (32):

Preferably, the fluoride source is present in an amount sufficient to provide from about 50 ppm to about 4,000 ppm fluoride ions in use. Inclusion of a fluoride source is beneficial, since fluoride ions are known to become incorporated into the hydroxyapatite of tooth enamel, thereby increasing the resistance of the enamel to decay. Fluoride is also now thought to act locally on the tooth enamel, altering the remineralisation-demineralisation balance in favour of reminderalisation. Inclusion of a fluoride source is also desirable when a polyphosphate anti-calculus agent is included, in order to inhibit the enzymic hydrolysis of such polyphosphates by salivary phosphatase enzymes.

Brief Summary Text (35):

Suitably, as described above, the compositions of the invention may include a further antimicrobial agent as a preservative and/or anti-plaque agent. Suitable antimicrobial agents include zinc salts (such as zinc citrate), cetyl pyridinium chloride, the bis-biguanides (such as chlorhexidine), aliphatic amines, bromochlorophene, hexachlorophene, salicylanilides, quaternary ammonium compounds and triclosan. Enzymic systems providing a source of a natural biocide may be used as alternatives to or in combination with the biocides listed. For example, a system comprising lactoperoxidase and glucose oxidase may be used to generate antimicrobial amounts of hydrogen peroxide in the presence of glucose, water and oxygen.

Brief Summary Text (40):

In a further aspect, the invention provides a method of treating or preventing dental caries or gingivitis by oral application of any oral hygiene composition as defined above.

Brief Summary Text (41):

In a further aspect, there is provided the use of any anti-microbial agent as defined above in the treatment or prevention of dental caries or gingivitis.

Brief Summary Text (42):

In a still further aspect, there is provided the use of any anti-microbial agent as defined above in the manufacture of a medicament for the treatment or prevention of dental caries or gingivitis.

Detailed Description Text (6):

An antimicrobial agent prepared substantially as described in Example 1 was tested for antimicrobial activity against Streptococcus salivarius and Streptococcus mutans, which are organisms known to be associated with dental caries/gingivitis and against Candida albicans.

Detailed Description Text (10):

These results show the activity of the antimicrobial agent of the present invention against organisms known to be associated with dental caries/gingivitis. It will be noted that activity was not detected in the control.

Detailed Description Text (13):

The anti-plaque activity of the compositions according to the present invention has been demonstrated as follows. Thin strips of aluminium were used as "artificial

tooth" surfaces on which plaque, collected from a small number of donors, was grown. Growth was encouraged by the provision of conditions resembling a normal oral environment (saliva, nutrients, pH and temperature) over a two day period with simulations made of the intake of two meals and of a sleeping, low nutrient period. The aluminium strips (and plaque) were exposed for one minute on two occasions to a suspension of the composition to be tested with distilled water and fresh saliva, thus simulating evening and morning toothbrushing sessions. Following the second exposure, plaque remaining on the strips after a four hour growth period and subsequent rinsing was dispersed by ultrasonic vibration. The optical density of the resulting plaque suspensions at 570 nm (two replicate readings per strip) was used to estimate the percentage reduction in plaque growth compared to plaque growth on test strips exposed to a control composition of saliva and water.

Detailed Description Text (34):
Antimicrobial tooth paint base

Detailed Description Text (35):
A tooth paint base is prepared in conventional manner to the following composition:

CLAIMS:

1. An oral composition suitable for use in oral hygiene or dental treatment comprising an effective amount of an antimicrobial agent in the form of a particulate, non-ion-exchanging, zeolite-free, inert metal oxide, a sparingly soluble metal salt providing antimicrobial metal ions in use and a further material selected from the group consisting of humectants, gelling agents, abrasives, fluoride sources, desensitizing agents, flavorings, colorings, sweeteners, preservatives, structuring agents, bactericides, anti-tartar agents and anti-plaque agents.
3. A composition as claimed in claim 1, wherein the metal oxide is selected from the group consisting of titanium dioxide, aluminum oxide, zirconium oxide, hafnium oxide, niobium oxide, tantalum oxide, calcium hydroxyapatite, barium sulphate, and magnesium oxide.
4. A composition as claimed in claim 3, wherein the titanium dioxide is in at least one of anatase, rutile and brookite crystalline form.
11. A composition as claimed in claim 1 wherein the metal oxide is titanium dioxide having a primary crystal size of from about 0.005 to 5 micrometers.
12. A composition as claimed in claim 11 wherein the titanium dioxide has a primary crystal size of from about 0.01 to 1 micrometers.
17. A method of treating or preventing dental caries or gingivitis, comprising applying to a mouth of an individual an oral hygiene composition comprising an effective amount of an antimicrobial agent comprising a particulate, non-ion-exchanging, zeolite-free, inert metal oxide and a sparingly soluble metal salt providing antimicrobial metal ions in use.
20. A composition as claimed in claim 1, wherein the composition is selected from the group consisting of a dentrifice, mouthwash, tooth powder, chewing gum, lozenge, mouth spray, denture cleansing formulation, tooth paint and glass ionomer cement.

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L4: Entry 26 of 37

File: USPT

Jan 28, 1997

US-PAT-NO: 5597554

DOCUMENT-IDENTIFIER: US 5597554 A

TITLE: Oral hygiene system

DATE-ISSUED: January 28, 1997

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Wagner; Eugene C.	Mount Kisco	NY		

US-CL-CURRENT: 424/53; 424/49, 424/52, 424/57

CLAIMS:

Having thus described the invention, there is claimed as new and desired to be secured by Letters Patent:

1. A method of cleansing tooth surfaces while reducing dentin abrasion which occurs during tooth brushing with a conventional abrasive-containing toothpaste, which comprises the steps of:

(a) providing a sufficient amount of a conventional toothpaste and containing from 20% to 50% by weight of an abrasive agent selected from the group consisting of hydrated silica, aluminum hydroxide, calcium carbonate, and dicalcium phosphate dihydrate;

(b) providing a sufficient amount of non-abrasive, homogeneously dispersed dentifrice comprising from 0.2% to 15% by weight of a peroxide compound, based upon the weight of total dentifrices;

(c) providing a toothbrush having bristles, the bristles having ends;

(d) placing the amount of conventional toothpaste from step (a) and the amount of dentifrice from step (b) in strips on the bristle ends, the strip being in other than side by side relationship;

(e) admixing the conventional toothpaste from step (a) with the dentifrice from step (b) to accelerate the release of oxygen from the dentifrice; and

(f) brushing tooth surfaces with the admixture from step (e).

2. The method of claim 1 wherein the volume amount of conventional toothpaste in step (a) is substantially equal to the volume amount of dentifrice in step (b).

3. The method of claim 1 wherein the mixture in step (c) comprises from 1.3 to 2.5 grams of conventional toothpaste and from 0.7 to 1.5 grams of dentifrice.

4. The method of claim 1 wherein the conventional toothpaste also contains one or more compounds selected from the group consisting of sodium fluoride, sodium monofluorophosphate, tetrasodium pyrophosphate, tetrasodium phosphate, titanium dioxide, cellulose gum, sodium hydroxide, trisodiumphosphate, and sodium phosphate, in an amount sufficient to accelerate decomposition of the peroxide compound.

5. The method of claim 1 wherein step (f) includes the step of placing the bristles of the toothbrush in the oral cavity and brushing tooth and gum surfaces.

6. The method of claim 1 wherein the peroxide compound is selected from the group consisting of hydrogen peroxide, urea peroxide, carbamide peroxide, and calcium peroxide.

7. A method of oral hygiene which reduces dentin abrasion normally occurring during tooth brushing with a conventional abrasive-containing toothpaste, the method comprising the steps of:

placing a strip of conventional abrasive-containing toothpaste containing from 20% to 50% by weight of an abrasive agent selected from the group consisting of hydrated silica, aluminum hydroxide, calcium carbonate, and dicalcium phosphate dihydrate and a strip of a non-abrasive, aqueous, homogeneously dispersed dentifrice comprising from 0.2% to 15% by weight of peroxide compound based upon the weight of the total dentifrice on the bristle ends of a toothbrush with the strips placed in other than side by side relationship,

(b) admixing the toothpaste and dentrifrice; and

(c) brushing tooth surfaces with the admixture from step (b).

8. The method of claim 7 wherein in step (a) the volume amount of conventional toothpaste is substantially equal to the volume amount of dentifrice.

9. The method of claim 7 wherein the mixture in step (b) comprises from 1.3 to 2.5 grams of conventional toothpaste and from 0.7 to 1.5 grams of dentifrice.

10. The method of claim 7 wherein the conventional toothpaste also contains one or more compounds selected from the group consisting of sodium fluoride, sodium monofluorophosphate, tetrasodium pyrophosphate, tetrasodium phosphate, titanium dioxide, cellulose gum, sodium hydroxide, trisodium phosphate, and sodium phosphate, in an amount sufficient to accelerate decomposition of the peroxide compound.

11. The method of claim 7 wherein step (a) includes the step of providing a toothbrush and dispensing substantially equal volumes of dentifrice and toothpaste on the bristles of the toothbrush, and step (b) includes the step of placing the bristles of the toothbrush in the oral cavity and brushing tooth and gum surfaces.

12. The method of claim 7 wherein the peroxide compound is selected from the group consisting of hydrogen peroxide, urea peroxide, carbamide peroxide, and calcium peroxide.

13. The method of claim 1 wherein step (d) includes placing the strips in end-to-end relationship.

14. The method of claim 1 wherein step (d) included placing one of the strips on top of the other strip.

15. The method of oral hygiene which reduces dentin abrasion normally occurring during tooth brushing with a conventional abrasive-containing toothpaste in accordance with claim 7 wherein one of the strips is placed on top of the other strip.

16. The method of oral hygiene which reduces abrasion normal occurring during tooth brushing with a conventional abrasive containing toothpaste in accordance with claim 7 wherein the strips are placed in end-to-end relationship.

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L4: Entry 29 of 37

File: USPT

Mar 28, 1995

US-PAT-NO: 5401495

DOCUMENT-IDENTIFIER: US 5401495 A

TITLE: Teeth whitener

DATE-ISSUED: March 28, 1995

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Murayama; Ron	Laguna Niguel	CA		

US-CL-CURRENT: 424/49; 424/53

CLAIMS:

Having thus described the invention, I claim:

1. A three component system for whitening human teeth wherein each component is applied to the teeth in a sequential manner, said three component system comprising:

a first component comprising a conditioning mouth rinse capable of cleansing the surface of said teeth;

a second component comprising a viscous bleaching gel including an effective concentration of hydrogen peroxide for bleaching said teeth, said hydrogen peroxide present in an amount ranging from 1 to 10% by weight of the total gel; and

a third component comprising a viscous polishing composition including an abrasive substance for polishing said teeth and a pigmenting agent capable of imparting a white color to said teeth, whereby said conditioning rinse, said bleaching gel and said polishing composition are applied individually and seriatim to said teeth.

2. The system as set forth in claim 1 wherein said conditioning rinse comprises a dilute aqueous solution of acetic acid.

3. The system as set forth in claim 2 wherein said acetic acid is present in said rinse at a concentration of approximately 1.5 percent.

4. The system as set forth in claim 1 wherein said conditioning rinse further includes substances selected from the following: flavor enhancers, preservatives, surfactants and mixtures thereof.

5. The system as set forth in claim 4 wherein said flavor enhancers are selected from the group consisting of sweeteners and flavoring chemicals, said sweeteners are selected from the group consisting of saccharin, sodium saccharin, methyl salicylate, sorbitol and aspartame, said flavoring chemicals are selected from the group consisting of chemicals imparting the flavor of menthol, spearmint, wintergreen and cinnamon, said preservatives selected from the group consisting of sodium benzoate, and said surfactants are selected from the group consisting of polyoxyethylene (20) sorbitan monolaurate, sodium dodecyl sulfate, sodium lauryl sulfate and mixtures thereof.

6. The system as set forth in claim 1 wherein said effective concentration of said hydrogen peroxide in said gel is between 3 percent and 8 percent.
7. The system as set forth in claim 6 wherein said concentration of said hydrogen peroxide is 6 percent.
8. The system as set forth in claim 1 wherein said bleaching gel additionally comprises a gelling agent, a thickening agent, and a neutralizing agent.
9. The system as set forth in claim 8 wherein said gelling agent is selected from the group consisting of a copolymer of acrylic acid cross-linked with polyallyl sucrose, organic polymer acid colloids selected from the group consisting of polyuronic acids, carboxypolymethylene compounds, polyester resins containing three carboxyl groups, partially hydrolyzed polyacrylates, polymethacrylates, polyoxyethylenes, polypropylene copolymers and mixtures thereof.
10. The system as set forth in claim 9 wherein said gelling agent is a copolymer of acrylic acid cross-linked with approximately 0.75 percent to 1.5 percent polyallyl sucrose.
11. The system as set forth in claim 8 wherein said neutralizing agent is selected from the group consisting of sodium hydroxide, potassium hydroxide and triethanolamine.
12. The system as set forth in claim 8 wherein said thickening agent comprises non-ionic cellulose gums selected from the group consisting of hydroxyethyl cellulose, hydroxy-propyl cellulose and carboxymethyl cellulose.
13. The system as set forth in claim 1 wherein said abrasive substance is alumina silicate particles.
14. The system as set forth in claim 13 wherein said alumina silicate has a particle size between 5 and 15 microns.
15. The system as set forth in claim 1 wherein said pigmenting agent is titanium dioxide particles.
16. The system as set forth in claim 15 wherein said titanium dioxide has a particle size of approximately 0.1 to 0.2 microns.
17. The system as set forth in claim 1 wherein said viscous polishing composition additionally comprises substances selected from the group consisting of carriers, thickening agents, surfactants, preservatives, flavor enhancers and mixtures thereof.
18. The system as set forth in claim 17 wherein said carriers comprise glycerin and water, said thickening agents comprise non-ionic cellulose gums selected from the group consisting of hydroxyethyl cellulose, hydroxypropyl cellulose and carboxymethyl cellulose, said surfactants are selected from the group consisting of polyoxyethylene (20) sorbitan monolaurate, sodium dodecyl sulfate, sodium lauryl sulfate and mixtures thereof, said preservatives selected from the group consisting of sodium benzoate, and said flavor enhancers are selected from the group consisting of sweeteners and flavoring chemicals, said sweeteners are selected from the group consisting of saccharin, sodium saccharin, methyl salicylate, sorbitol and aspartame, and said flavoring chemicals are selected from the group consisting of chemicals imparting the flavor of menthol, spearmint, wintergreen and cinnamon.

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L4: Entry 32 of 37

File: USPT

Apr 12, 1994

US-PAT-NO: 5302374

DOCUMENT-IDENTIFIER: US 5302374 A

TITLE: Oral hygiene system

DATE-ISSUED: April 12, 1994

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Wagner; Eugene C.	Chappaqua	NY		

US-CL-CURRENT: 424/52; 424/49, 424/53

CLAIMS:

Having thus described the invention, there is claimed as new and desired to be secured by Letters Patent:

1. A method of cleansing tooth surfaces while reducing dentin abrasion which occurs during tooth brushing with a conventional abrasive-containing toothpaste, which comprises the steps of:

(a) providing a sufficient amount of a conventional toothpaste containing from 20% to 50% by weight of an abrasive agent selected from the group consisting of hydrated silica, aluminum hydroxide, calcium carbonate, and dicalcium phosphate dihydrate;

(b) providing a sufficient amount of non-abrasive, aqueous, homogeneously dispersed dentifrice having a toothpaste consistency and comprising from 0.2% to 10% by weight of hydrogen peroxide, from 0.2% to 4% by weight of Carbomer 940 gelling agent, from 2% to 35% by weight of corn starch filler, and from 0.2% to 2% by weight of surfactant and foaming agent, based upon the weight of the total dentifrice;

(c) admixing the conventional toothpaste from step (a) with the dentifrice from step (b) to accelerate the release of oxygen from the dentifrice; and

(d) brushing tooth surfaces with the admixture from step (c).

2. The method of claim 1, wherein the volume amount of conventional toothpaste in step (a) is substantially equal to the volume amount of dentifrice in step (b).

3. The method of claim 1, wherein the mixture in step (c) comprises from 1.3 to 2.5 grams of conventional toothpaste and from 0.7 to 1.5 grams of dentifrice.

4. The method of claim 1, wherein the conventional toothpaste also contains one or more compounds selected from the group consisting of sodium fluoride, sodium monofluorophosphate, tetrasodium pyrophosphate, tetrasodium phosphate, titanium dioxide, cellulose gum, sodium hydroxide, trisodium phosphate, and sodium phosphate, in an amount sufficient to accelerate decomposition of the hydrogen peroxide.

5. The method of claim 1, wherein step (c) includes the step of providing a

toothbrush and dispensing substantially equal volumes of dentifrice and toothpaste on the bristles of the toothbrush, and step (d) includes the step of placing the bristles of the toothbrush in the oral cavity and brushing tooth and gum surfaces.

6. A method of cleansing tooth surfaces while reducing dentin abrasion which occurs during tooth brushing with a conventional abrasive-containing toothpaste, which comprises the steps of:

(a) admixing an amount of conventional abrasive-containing toothpaste containing from 20% to 50% by weight of an abrasive agent selected from the group consisting of hydrated silica, aluminum hydroxide, calcium carbonate, and dicalcium phosphate dihydrate with an amount of a non-abrasive, aqueous, homogeneously dispersed dentifrice having a toothpaste consistency and comprising from 0.2% to 10% by weight of hydrogen peroxide, from 0.25% to 4% by weight of Carbomer 940 gelling agent, from 2% to 35% by weight of corn starch filler, and from 0.2% to 2% by weight of surfactant and foaming agent, based upon the weight of the total dentifrice; and

(b) brushing tooth surfaces with the admixture from step (a).

7. The method of claim 6, wherein the step (a) the volume amount of conventional toothpaste is substantially equal to the volume amount of dentifrice.

8. The method of claim 6, wherein the mixture in step (c) comprises from 1.3 to 2.5 grams of conventional toothpaste and from 0.7 to 1.5 grams of dentifrice.

9. The method of claim 6, wherein the conventional toothpaste also contains one or more compounds selected from the group consisting of sodium fluoride, sodium monofluorophosphate, tetrasodium pyrophosphate, tetrasodium phosphate, titanium dioxide, cellulose gum, sodium hydroxide, trisodium phosphate, and sodium phosphate, in an amount sufficient to accelerate decomposition of the hydrogen peroxide.

10. The method of claim 6, wherein step (c) includes the step of providing a toothbrush and dispensing substantially equal volumes of dentifrice and toothpaste on the bristles of the toothbrush, and step (d) includes the step of placing the bristles of the toothbrush in the oral cavity and brushing tooth and gum surfaces.

11. A dentifrice-toothpaste mixture suitable for instantaneous employment in an oral hygiene program for the treatment of tooth surfaces and gum tissue, the mixture comprising (a) a non-abrasive, aqueous, homogeneously dispersed dentifrice having a toothpaste consistency and comprising from 0.2% to 10% by weight of hydrogen peroxide, from 0.25% to 4% by weight of Carbomer 940 gelling agent, from 2% to 35% by weight of corn starch filler, and from 0.2% to 2% by weight of surfactant and foaming agent, based upon the weight of the total dentifrice, and (b) a conventional abrasive-containing toothpaste containing from 20% to 50% by weight of an abrasive agent selected from the group consisting of hydrated silica, aluminum hydroxide, calcium carbonate, and dicalcium phosphate dihydrate, the abrasive agent serving to accelerate the decomposition of the hydrogen peroxide in the dentifrice upon formation of the mixture.

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L4: Entry 33 of 37

File: USPT

Jun 16, 1992

US-PAT-NO: 5122365

DOCUMENT-IDENTIFIER: US 5122365 A

TITLE: Teeth whitener

DATE-ISSUED: June 16, 1992

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Murayama; Ron	Laguna Niguel	CA		

US-CL-CURRENT: 424/49; 252/186.3, 424/52, 424/53

CLAIMS:

Having thus described the invention, I claim:

1. A three component package for whitening human teeth wherein each component is applied to the teeth in a sequential manner, said three component package comprising:

a first component comprising a conditioning mouth rinse capable of cleansing the surface of said teeth, said mouth rinse comprising an aqueous solution of acetic acid; and

a second component comprising a viscous bleaching gel including an effective concentration of hydrogen peroxide for bleaching said teeth, said hydrogen peroxide present in an amount ranging from 1 to 10% by weight of the total gel; and

a third component comprising a viscous polishing composition including an abrasive substance comprising alumina silicates for polishing said teeth and a pigmenting agent capable of imparting a white color to said teeth, whereby said conditioning rinse, said bleaching gel and said polishing composition are applied individually and seriatim to said teeth.

2. The package as set forth in claim 1 wherein said conditioning rinse comprises a dilute aqueous solution of acetic acid.

3. The package as set forth in claim 2 wherein said acetic acid is present in said rinse at a concentration of approximately 1.5 percent.

4. The package as set forth in claim 1 wherein said conditioning rinse further includes substances selected from the group consisting of flavor enhancers, preservatives, surfactants and mixtures thereof.

5. The package as set forth in claim 4 wherein said flavor enhancers are selected from the group consisting of sweeteners and flavoring chemicals, said sweeteners are selected from the group consisting of saccharin, sodium saccharin, methyl salicylate, sorbitol and aspartame, said flavoring chemicals are selected from the group consisting of chemicals imparting the flavor of menthol, spearmint, wintergreen and cinnamon, said preservatives including sodium benzoate, and said surfactants are selected from the group consisting of polyoxyethylene (20) sorbitan monolaurate sodium dodecyl sulfate, sodium lauryl

sulfate and mixtures thereof.

6. The package as set forth in claim 1 wherein said effective concentration of said hydrogen peroxide in said gel is between 3 percent and 8 percent.

7. The package as set forth in claim 6 wherein said concentration of said hydrogen peroxide is 6 percent.

8. The package as set forth in claim 1 wherein said bleaching gel additionally comprises a gelling agent, a thickening agent, and a neutralizing agent.

9. The package as set forth in claim 8 wherein said gelling agent is selected from the group consisting of a copolymer of acrylic acid cross-linked with polyallyl sucrose, organic polymer acid colloids including polyuronic acids, carboxypolymethylene compounds, polyester resins containing three carboxyl groups, partially hydrolyzed polyacrylates, polymethacrylates, polyoxyethylenes, polypropylene copolymers and mixtures thereof.

10. The package as set forth in claim 9 wherein said gelling agent is a copolymer of acrylic acid cross-linked with approximately 0.75 percent to 1.5 percent polyallyl sucrose.

11. The package system as set forth in claim 8 wherein said neutralizing agent is selected from the group consisting of sodium hydroxide, potassium hydroxide and triethanolamine.

12. The package as set forth in claim 8 wherein said thickening agent comprises non-ionic cellulose gums selected from the group consisting of hydroxyethyl cellulose, hydroxypropyl cellulose and carboxymethyl cellulose.

13. The package as set forth in claim 1 wherein said viscous polishing composition additionally comprises substances selected from the group consisting of carriers, thickening agents, surfactants, preservatives, flavor enhancers and mixtures thereof.

14. The package as set forth in claim 1 wherein said alumina silicate has a particle size between 5 and 15 microns.

15. The package as set forth in claim 1 wherein said pigmenting agent is titanium dioxide particles.

16. The package as set forth in claim 15 wherein said titanium dioxide has a particle size of approximately 0.1 to 0.2 microns.

17. The package as set forth in claim 13 wherein said carriers comprise glycerin and water, said thickening agents comprise non-ionic cellulose gums selected from the group consisting of hydroxyethyl cellulose, hydroxypropyl cellulose and carboxymethyl cellulose, said surfactants are selected from the group consisting of polyoxyethylene (20) sorbitan monolaurate Tween-20, sodium dodecyl sulfate, sodium lauryl sulfate and mixtures thereof, said preservatives include sodium benzoate, and said flavor enhancers are selected from the group consisting of sweeteners and flavoring chemicals, said sweeteners are selected from the group consisting of saccharin, sodium saccharin, methyl salicylate, sorbitol and aspartame, and said flavoring chemicals are selected from the group consisting of chemicals imparting the flavor of menthol, spearmint, wintergreen and cinnamon.

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L5: Entry 44 of 45

File: USPT

Dec 10, 1974

US-PAT-NO: 3853575

DOCUMENT-IDENTIFIER: US 3853575 A

TITLE: SUBSTANTIALLY NON-GREYING TITANIUM DIOXIDE PIGMENTS FOR USE IN RESIN COMPOSITIONS

DATE-ISSUED: December 10, 1974

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Holle; Bernd	Krefeld			DT
Winter; Gerhard	Krefeld			DT

US-CL-CURRENT: 106/442

CLAIMS:

What is claimed is:

1. A process for the production of a substantially non-greying titanium dioxide pigment coated with a layer of aluminum oxide aquate, comprising drying at about 25.degree. to 200.degree.C a suspension of a titanium dioxide pigment in a solution of a basic aluminum salt, said solution being selected from the group consisting of (a) a solution of basic aluminum chloride containing an oxidizing salt, (b) a solution of basic aluminum nitrate, and (c) mixtures thereof, the basic aluminum salt being present in said solution in such amount that the dried titanium dioxide pigment contains about 0.5 to about 10% by weight of aluminum oxide aquate (calculated as Al.sub.2 O.sub.3), and the nitrate and oxidizing salts being present in such amount in the solution that their anions are present in the titanium dioxide pigment to the extent of about 0.05 to 3% by weight.
2. The process of claim 1, wherein the basic aluminum salt has a ratio of aluminum cation Al.sub.3.sup.+ to chloride or nitrate anion of about 0.5 to 5.
3. The process of claim 2, wherein the basic aluminum salt has a ratio of aluminum cation (Al.sub.3.sup.+) to chloride or nitrate anion of about 1 to 3.
4. The process of claim 2, wherein the basic aluminum salt has the approximate composition $\text{Al.sub.2 (OH).sub.5 Cl}$.
5. The process of claim 3, wherein the basic aluminum salt has the approximate composition $\text{Al(OH).sub.2.5 (NO.sub.3).sub.0.5}$.
6. The process of claim 1, wherein the solution of the basic aluminum salt is a solution of basic aluminum chloride containing an oxidizing salt selected from the group consisting of a nitrate, nitrite, peroxy disulfate, peroxidic addition compound or mixture thereof.
7. The process of claim 1, wherein the solution of the basic aluminum salt is a solution of basic aluminum chloride containing $\text{NaB0.sub.2 . H.sub.2 O.sub.2}$
8. The process of claim 1, wherein the suspension is dried at about 80.degree. to 120.degree.C, the basic aluminum salt having a ratio of aluminum cation Al.sub.3.sup.+ to chloride or nitrate anion of from about 1 to 3 and being

present in such amount that the dried titanium dioxide pigment contains about 2 to 5% by weight of aluminum oxide aquate (calculated as Al.sub.2 O.sub.3) the nitrate and oxidizing salts being present in an amount such that their anions are present in about 0.2 to 2% by weight of the titanium dioxide pigment.

9. A process for the production of a substantially non-greying titanium dioxide pigment coated with a layer of aluminum oxide aquate, comprising adding a solution of basic aluminum chloride or nitrate to a titanium dioxide pigment suspension, adjusting the pH to about 5 or 6, adding an aqueous solution of an oxidizing salt, adjusting the pH to about 6 to 7 initially, subsequently adjusting the pH to about 7 to 8, filtering and then washing the pigment with water, and drying at about 100.degree.C to 200.degree.C, the basic aluminum chloride or nitrate being present in said suspension in such amount that the dried titanium dioxide pigment contains about 0.5 to about 10% by weight of aluminum oxide aquate (calculated as Al.sub.2 O.sub.3), and the oxidizing salt and aluminum nitrate, if the latter is present, being present in such amount in the suspension that their anions are present in the titanium dioxide pigment to the extent of about 0.05 to 3% by weight.

10. A titanium dioxide pigment coated with about 0.5 to 10% by weight of aluminum oxide aquate (calculated as Al.sub.2 O.sub.3) and about 0.05 to 3% by weight of an anion of an oxidizing salt selected from the group consisting of a nitrate, nitrite, peroxy disulfate, perborate or mixture thereof.

11. A titanium dioxide pigment according to claim 10, wherein the aluminum oxide aquate is present in about 2 to 5% (calculated as Al.sub.2 O.sub.3) and the anion of an oxidizing salt is present in about 0.2 to 2% by weight.

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File: USPT

Dec 10, 1974

DOCUMENT-IDENTIFIER: US 3853575 A

TITLE: SUBSTANTIALLY NON-GREYING TITANIUM DIOXIDE PIGMENTS FOR USE IN RESIN COMPOSITIONS

CLAIMS:

1. A process for the production of a substantially non-greying titanium dioxide pigment coated with a layer of aluminum oxide aquate, comprising drying at about 25.degree. to 200.degree.C a suspension of a titanium dioxide pigment in a solution of a basic aluminum salt, said solution being selected from the group consisting of (a) a solution of basic aluminum chloride containing an oxidizing salt, (b) a solution of basic aluminum nitrate, and (c) mixtures thereof, the basic aluminum salt being present in said solution in such amount that the dried titanium dioxide pigment contains about 0.5 to about 10% by weight of aluminum oxide aquate (calculated as Al.sub.2 O.sub.3), and the nitrate and oxidizing salts being present in such amount in the solution that their anions are present in the titanium dioxide pigment to the extent of about 0.05 to 3% by weight.
8. The process of claim 1, wherein the suspension is dried at about 80.degree. to 120.degree.C, the basic aluminum salt having a ratio of aluminum cation Al.sup.3.sup.+) to chloride or nitrate anion of from about 1 to 3 and being present in such amount that the dried titanium dioxide pigment contains about 2 to 5% by weight of aluminum oxide aquate (calculated as Al.sub.2 O.sub.3) the nitrate and oxidizing salts being present in an amount such that their anions are present in about 0.2 to 2% by weight of the titanium dioxide pigment.
9. A process for the production of a substantially non-greying titanium dioxide pigment coated with a layer of aluminum oxide aquate, comprising adding a solution of basic aluminum chloride or nitrate to a titanium dioxide pigment suspension, adjusting the pH to about 5 or 6, adding an aqueous solution of an oxidizing salt, adjusting the pH to about 6 to 7 initially, subsequently adjusting the pH to about 7 to 8, filtering and then washing the pigment with water, and drying at about 100.degree.C to 200.degree.C, the basic aluminum chloride or nitrate being present in said suspension in such amount that the dried titanium dioxide pigment contains about 0.5 to about 10% by weight of aluminum oxide aquate (calculated as Al.sub.2 O.sub.3), and the oxidizing salt and aluminum nitrate, if the latter is present, being present in such amount in the suspension that their anions are present in the titanium dioxide pigment to the extent of about 0.05 to 3% by weight.
10. A titanium dioxide pigment coated with about 0.5 to 10% by weight of aluminum oxide aquate (calculated as Al.sub.2 O.sub.3) and about 0.05 to 3% by weight of an anion of an oxidizing salt selected from the group consisting of a nitrate, nitrite, peroxy disulfate, perborate or mixture thereof.
11. A titanium dioxide pigment according to claim 10, wherein the aluminum oxide aquate is present in about 2 to 5% (calculated as Al.sub.2 O.sub.3) and the anion of an oxidizing salt is present in about 0.2 to 2% by weight.

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L5: Entry 42 of 45

File: USPT

May 13, 1975

US-PAT-NO: 3883373

DOCUMENT-IDENTIFIER: US 3883373 A

TITLE: Gas generating compositions

DATE-ISSUED: May 13, 1975

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Sidebottom; Eric William	Otterburn Heights, Quebec			CA

US-CL-CURRENT: 149/6; 149/35, 149/37, 149/40, 149/41, 149/42, 149/43, 149/45,
149/75, 149/77, 423/351

CLAIMS:

What we claim is:

1. A gas generating composition comprising a mixture of particles of the following ingredients

1. an alkali metal azide or an alkaline earth metal azide,

2. an oxidizing compound in proportion sufficient to react completely with said azide with the liberation of nitrogen therefrom, and

3. an oxide selected from the group consisting of silicon dioxide, aluminum oxide, titanium dioxide, tin oxide and zinc oxide, with or without admixture with a metal selected from the group consisting of silicon, aluminum, tin and zinc, said oxide and metal being in proportion sufficient to react with all the metallic residue of the reaction between (1) and (2).

2. A gas generating composition as claimed in claim 1 wherein the azide is selected from the group consisting of lithium azide, sodium azide, potassium azide, rubidium azide, cesium azide, calcium azide, magnesium azide, strontium azide and barium azide.

3. A gas generating composition as claimed in claim 1 wherein the oxidizing compound is a metal peroxide selected from the group consisting of sodium peroxide, potassium peroxide, rubidium peroxide, cesium peroxide, calcium peroxide, strontium peroxide and barium peroxide.

4. A gas generating composition as claimed in claim 1 wherein the oxidizing compound is an inorganic perchlorate selected from the group consisting of lithium perchlorate, sodium perchlorate, potassium perchlorate, rubidium perchlorate, magnesium perchlorate, calcium perchlorate, strontium perchlorate, barium perchlorate, ferric perchlorate and cobalt perchlorate.

5. A gas generating composition as claimed in claim 1 wherein the oxidizing compound is a metal nitrate selected from the group consisting of lithium nitrate, sodium nitrate, potassium nitrate, copper nitrate, silver nitrate, magnesium nitrate, barium nitrate, zinc nitrate, aluminum nitrate, thallium nitrate, stannic nitrate, bismuth nitrate, manganese nitrate, ferric nitrate, ferrous nitrate and nickel nitrate.

6. A gas generating composition as claimed in claim 1 including as ingredient fumed silica coated with a water repellent.

7. A gas generating composition as claimed in claim 6 wherein the water repellent is a silane.

8. A gas generating composition as claimed in claim 6 wherein the fumed silica ingredient comprises 2% by weight of the composition.

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L5: Entry 36 of 45

File: USPT

Sep 1, 1987

US-PAT-NO: 4690776

DOCUMENT-IDENTIFIER: US 4690776 A

TITLE: Method of manufacture of a toothpaste composition

DATE-ISSUED: September 1, 1987

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Smigel; Irwin E.	New York	NY	10022	

US-CL-CURRENT: 424/49; 424/44, 424/53, 424/57, 514/835

CLAIMS:

What is claimed is:

1. A method of preparing a toothpaste composition comprising adding calcium phosphate in an amount of 0.5 to 5% by weight of the composition and sodium perborate in an amount of 0.5 to 5% by weight of the composition to hot water to form a first mixture and agitating the first mixture, adding to said first mixture sorbitol in an amount of 1 to 50% by weight of the composition, cornstarch in an amount of 0.5 to 10% by weight of the composition and aluminum hydroxide in an amount of 0.01 to 1% by weight of the composition to form a second mixture and agitating the second mixture, adding to said second mixture dicalcium phosphate in an amount of 1 to 50% by weight of the composition and sodium monofluoride phosphate in an amount of 0.7 to 0.8% by weight of the composition to form a third mixture and agitating the third mixture, adding to the third mixture sodium bicarbonate, in an amount of 1 to 50% by weight of the composition, gradually adding increments of a flavoring material in an amount of 0.05 to 2% to control any foaming and to facilitate release of gases to form a fourth mixture, and adding to the fourth mixture sodium lauryl sulfoacetate in an amount of 0.1 to 5% and gum in an amount of 0.5 to 5% and agitating the fourth mixture until a homogeneous paste composition is obtained.
2. A method as claimed in claim 1 wherein the water is initially heated to 150.degree. F.
3. A method as claimed in claim 2 wherein the first, second and third mixtures are each agitated for ten minutes.
4. A method as claimed in claim 2 comprising adding an alkylparaben in an amount of 0.05 to 1.0% by weight of the composition to the second mixture before agitation thereof.
5. A method as claimed in claim 2 comprising adding calcium carbonate in an amount of 1 to 50% by weight of the composition and magnesium carbonate in an amount of 1 to 25% by weight of the composition to the third mixture before agitation thereof.
6. A method as claimed in claim 2 comprising adding to the fourth mixture titanium dioxide as a whitening agent in an amount of 0.1 to 10% by weight of the composition and saccharinate in an amount of from 0.05 to 2% by weight of the composition and the resulting mixture is agitated for at least one half

hour.

7. A method as claimed in claim 2 comprising adding a cellulose gum in an amount of 0.5 to 5% by weight of the composition to the fourth mixture before agitation thereof.

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File: USPT

Sep 1, 1987

DOCUMENT-IDENTIFIER: US 4690776 A

TITLE: Method of manufacture of a toothpaste composition

CLAIMS:

1. A method of preparing a toothpaste composition comprising adding calcium phosphate in an amount of 0.5 to 5% by weight of the composition and sodium perborate in an amount of 0.5 to 5% by weight of the composition to hot water to form a first mixture and agitating the first mixture, adding to said first mixture sorbitol in an amount of 1 to 50% by weight of the composition, cornstarch in an amount of 0.5 to 10% by weight of the composition and aluminum hydroxide in an amount of 0.01 to 1% by weight of the composition to form a second mixture and agitating the second mixture, adding to said second mixture dicalcium phosphate in an amount of 1 to 50% by weight of the composition and sodium monofluoride phosphate in an amount of 0.7 to 0.8% by weight of the composition to form a third mixture and agitating the third mixture, adding to the third mixture sodium bicarbonate, in an amount of 1 to 50% by weight of the composition, gradually adding increments of a flavoring material in an amount of 0.05 to 2% to control any foaming and to facilitate release of gases to form a fourth mixture, and adding to the fourth mixture sodium lauryl sulfoacetate in an amount of 0.1 to 5% and gum in an amount of 0.5 to 5% and agitating the fourth mixture until a homogeneous paste composition is obtained.

6. A method as claimed in claim 2 comprising adding to the fourth mixture titanium dioxide as a whitening agent in an amount of 0.1 to 10% by weight of the composition and saccharinate in an amount of from 0.05 to 2% by weight of the composition and the resulting mixture is agitated for at least one half hour.

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L5: Entry 20 of 45

File: USPT

Jan '28, 1997

US-PAT-NO: 5597554

DOCUMENT-IDENTIFIER: US 5597554 A

TITLE: Oral hygiene system

DATE-ISSUED: January 28, 1997

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Wagner; Eugene C.	Mount Kisco	NY		

US-CL-CURRENT: 424/53; 424/49, 424/52, 424/57

CLAIMS:

Having thus described the invention, there is claimed as new and desired to be secured by Letters Patent:

1. A method of cleansing tooth surfaces while reducing dentin abrasion which occurs during tooth brushing with a conventional abrasive-containing toothpaste, which comprises the steps of:

(a) providing a sufficient amount of a conventional toothpaste and containing from 20% to 50% by weight of an abrasive agent selected from the group consisting of hydrated silica, aluminum hydroxide, calcium carbonate, and dicalcium phosphate dihydrate;

(b) providing a sufficient amount of non-abrasive, homogeneously dispersed dentifrice comprising from 0.2% to 15% by weight of a peroxide compound, based upon the weight of total dentifrices;

(c) providing a toothbrush having bristles, the bristles having ends;

(d) placing the amount of conventional toothpaste from step (a) and the amount of dentrifrice from step (b) in strips on the bristle ends, the strip being in other than side by side relationship;

(e) admixing the conventional toothpaste from step (a) with the dentifrice from step (b) to accelerate the release of oxygen from the dentifrice; and

(f) brushing tooth surfaces with the admixture from step (e).

2. The method of claim 1 wherein the volume amount of conventional toothpaste in step (a) is substantially equal to the volume amount of dentifrice in step (b).

3. The method of claim 1 wherein the mixture in step (c) comprises from 1.3 to 2.5 grams of conventional toothpaste and from 0.7 to 1.5 grams of dentifrice.

4. The method of claim 1 wherein the conventional toothpaste also contains one or more compounds selected from the group consisting of sodium fluoride, sodium monofluorophosphate, tetrasodium pyrophosphate, tetrasodium phosphate, titanium dioxide, cellulose gum, sodium hydroxide, trisodiumphosphate, and sodium phosphate, in an amount sufficient to accelerate decomposition of the peroxide compound.

5. The method of claim 1 wherein step (f) includes the step of placing the bristles of the toothbrush in the oral cavity and brushing tooth and gum surfaces.

6. The method of claim 1 wherein the peroxide compound is selected from the group consisting of hydrogen peroxide, urea peroxide, carbamide peroxide, and calcium peroxide.

7. A method of oral hygiene which reduces dentin abrasion normally occurring during tooth brushing with a conventional abrasive-containing toothpaste, the method comprising the steps of:

placing a strip of conventional abrasive-containing toothpaste containing from 20% to 50% by weight of an abrasive agent selected from the group consisting of hydrated silica, aluminum hydroxide, calcium carbonate, and dicalcium phosphate dihydrate and a strip of a non-abrasive, aqueous, homogeneously dispersed dentifrice comprising from 0.2% to 15% by weight of peroxide compound based upon the weight of the total dentifrice on the bristle ends of a toothbrush with the strips placed in other than side by side relationship,

(b) admixing the toothpaste and dentifrice; and

(c) brushing tooth surfaces with the admixture from step (b).

8. The method of claim 7 wherein in step (a) the volume amount of conventional toothpaste is substantially equal to the volume amount of dentifrice.

9. The method of claim 7 wherein the mixture in step (b) comprises from 1.3 to 2.5 grams of conventional toothpaste and from 0.7 to 1.5 grams of dentifrice.

10. The method of claim 7 wherein the conventional toothpaste also contains one or more compounds selected from the group consisting of sodium fluoride, sodium monofluorophosphate, tetrasodium pyrophosphate, tetrasodium phosphate, titanium dioxide, cellulose gum, sodium hydroxide, trisodium phosphate, and sodium phosphate, in an amount sufficient to accelerate decomposition of the peroxide compound.

11. The method of claim 7 wherein step (a) includes the step of providing a toothbrush and dispensing substantially equal volumes of dentifrice and toothpaste on the bristles of the toothbrush, and step (b) includes the step of placing the bristles of the toothbrush in the oral cavity and brushing tooth and gum surfaces.

12. The method of claim 7 wherein the peroxide compound is selected from the group consisting of hydrogen peroxide, urea peroxide, carbamide peroxide, and calcium peroxide.

13. The method of claim 1 wherein step (d) includes placing the strips in end-to-end relationship.

14. The method of claim 1 wherein step (d) included placing one of the strips on top of the other strip.

15. The method of oral hygiene which reduces dentin abrasion normally occurring during tooth brushing with a conventional abrasive-containing toothpaste in accordance with claim 7 wherein one of the strips is placed on top of the other strip.

16. The method of oral hygiene which reduces abrasion normal occurring during tooth brushing with a conventional abrasive containing toothpaste in accordance with claim 7 wherein the strips are placed in end-to-end relationship.

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L5: Entry 19 of 45

File: USPT

Dec 16, 1997

US-PAT-NO: 5698205

DOCUMENT-IDENTIFIER: US 5698205 A

TITLE: Photostabilization of titanium dioxide sols

DATE-ISSUED: December 16, 1997

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Bruckner; Hans-Dieter	Darmstadt			DE
Hamann; Ulrike	Darmstadt			DE
Heyland; Andrea	Reichelsheim			DE

US-CL-CURRENT: 424/401; 252/363.5, 423/610, 423/612, 424/59, 424/70.9, 516/90,
516/905, 516/918, 516/919

CLAIMS:

What is claimed is:

1. A titanium dioxide sol comprising:titanium dioxide sol particles,

an aqueous dispersion medium,

5-40 mole %, based on TiO.sub.2 content of sol, of a hydroxycarboxylic acid or a salt, ester or amide thereof as a stabilizing agent, and

0.1-20 mole %, based on TiO.sub.2 content of sol, of a further stabilizing agent, wherein said further stabilizing agent is selected from the group consisting of La.sup.2+, Fe.sup.2+, Fe.sup.3+, Sn.sup.4+, Zn.sup.2+, Ce.sup.3+, Mn.sup.2+, inorganic fluorides, inorganic iodates, inorganic nitrates, one or more complexing agents, one or more oxidizing agents, and combinations thereof,

wherein said sol is photostable and neutral.

2. A photostable titanium dioxide sol according to claim 1, wherein said hydroxycarboxylic acid is citric acid or tartaric acid.3. A photostable titanium dioxide sol according to claim 1, wherein said further stabilizing agent is selected from the group consisting of La.sup.2+, Zn.sup.2+, Sn.sup.4+, Mn.sup.2+, Ce.sup.3+, Fe.sup.2+ and Fe.sup.3+.4. A photostable titanium dioxide sol according to claim 1, wherein said further stabilizing agent is selected from the group consisting of [F.sup.-, IO.sub.3.sup.-, SO.sub.4.sup.2- and NO.sub.3.sup.-] inorganic fluorides, inorganic iodates and inorganic nitrates.5. A photostable titanium dioxide sol according to claim 1, wherein said further stabilizing agent is EDTA.6. A photostable titanium dioxide sol according to claim 1, wherein said further stabilizing agent is a perchlorate or a peroxodisulfate.

7. A photostable titanium dioxide sol according to claim 1, wherein said hydroxycarboxylic acid is citric acid or tartaric acid; said complexing agent is EDTA; and said oxidizing agents are perchlorates and peroxodisulfates.

8. A titanium dioxide sol according to claim 1, wherein said oxidizing agents are hydrogen peroxide, perchlorates, perborates and peroxodisulfates.

9. A process for the preparation of a photostable titanium dioxide sol, said sol comprising titanium dioxide sol particles, and an aqueous dispersion medium, said process comprising:

stabilizing said sol by adding 5-40 mole %, based on TiO_2 content of said sol, of a hydroxycarboxylic acid or a salt, ester or amide thereof as a stabilizing agent, and

after stabilization of said sol with said hydroxycarboxylic acid or a salt, ester or amide thereof, said sol having a pH of 6-8, adding 0.1-20 mole %, based on TiO_2 content of said sol, of a further stabilizing agent selected from the group consisting of metal ions, anions, one or more complexing agents, one or more oxidizing agents, and combinations thereof.

10. A process for preparing a dried photostable titanium dioxide sol, comprising:

drying a sol prepared according to claim 9, and further comprising drying said sol.

11. A process according to claim 10, further comprising precipitating said sol with an alcohol prior to drying.

12. A process for precipitating a photostable titanium dioxide sol comprising: adding an alcohol to a sol prepared according to claim 9.

13. In a cosmetic formulation containing a carrier and a titanium dioxide sol particle, the improvement wherein said formulation contains a titanium dioxide sol prepared according to claim 9.

14. A process according to claim 9, wherein said metal ions are La^{2+} , Fe^{2+} , Fe^{3+} , Sn^{4+} , Zn^{2+} , Ce^{3+} and Mn^{2+} , and said anions are inorganic fluorides, inorganic iodates and inorganic nitrates.

15. A process according to claim 14, wherein said one or more complexing agents are EDTA, nitrilotriacetic acid, 1,2-diaminocyclohexylene-dinitrilotetraacetic acid, diethylenetriamine pentaacetic acid, N-(2-hydroxy-ethyl)-ethylenediamino-N,N,N-triacetic acid and tetraethylenetetraaminohexaacetic acid, and said one or more oxidizing agents are perchlorates and peroxodisulfates.

16. A process according to claim 9, wherein said oxidizing agents are hydrogen peroxide, perchlorates, perborates and peroxodisulfates.

17. A titanium dioxide sol comprising:

titanium dioxide sol particles having a particle size of 5-200 nm,

an aqueous dispersion medium,

5-40 mole %, based on TiO_2 content of sol, of a stabilizing agent selected from the group consisting of hydroxy carboxylic acid having 1-6 hydroxy groups and 1-4--COOH groups, a monoalkyl amide thereof wherein the alkyl group has 1-10 C atoms, a dialkyl amide thereof wherein each, alkyl group has 1-10 C atoms, an alkali metal salt thereof, an alkaline earth salt thereof, and an amine salt thereof, and

0.1-20 mole %, based on TiO_2 content of sol, of a further stabilizing agent, wherein said further stabilizing agent is at least one metal ion, at least one anion, at least one complexing agent, at least one oxidizing agent, or combinations thereof,

wherein said at least one metal ion is La^{2+} , Zn^{2+} , Sn^{4+} , Mn^{2+} , Ce^{3+} , Fe^{2+} , Fe^{3+} , or combinations thereof,

wherein said at least one anion are an inorganic fluoride, an inorganic iodate, an inorganic nitrate or combinations thereof,

wherein said at least one complexing agent is EDTA, nitrilotriacetic acid, 1,2-di-aminocyclohexylene-dinitrilotetraacetic acid, diethylenetriaminepentaacetic acid, N-(2-hydroxy-ethyl)-ethylenediamino-N,N,N-triacetic acid, tetraethylenetetraaminohexaacetic acid or combinations thereof, and

wherein said at least one oxidizing agent is an inorganic peroxide or combinations thereof.

18. A titanium dioxide sol according to claim 17, wherein said at least one oxidizing agent is hydrogen peroxide, a perchlorate, a perborate, a peroxodisulfate or combinations thereof.

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File: USPT

Dec 16, 1997

DOCUMENT-IDENTIFIER: US 5698205 A

TITLE: Photostabilization of titanium dioxide sols

CLAIMS:

1. A titanium dioxide sol comprising:

titanium dioxide sol particles,

an aqueous dispersion medium,

5-40 mole %, based on TiO_2 content of sol, of a hydroxycarboxylic acid or a salt, ester or amide thereof as a stabilizing agent, and

0.1-20 mole %, based on TiO_2 content of sol, of a further stabilizing agent, wherein said further stabilizing agent is selected from the group consisting of La^{3+} , Fe^{2+} , Fe^{3+} , Sn^{4+} , Zn^{2+} , Ce^{3+} , Mn^{2+} , inorganic fluorides, inorganic iodates, inorganic nitrates, one or more complexing agents, one or more oxidizing agents, and combinations thereof,

wherein said sol is photostable and neutral.

2. A photostable titanium dioxide sol according to claim 1, wherein said hydroxycarboxylic acid is citric acid or tartaric acid.

3. A photostable titanium dioxide sol according to claim 1, wherein said further stabilizing agent is selected from the group consisting of La^{3+} , Zn^{2+} , Sn^{4+} , Mn^{2+} , Ce^{3+} , Fe^{2+} and Fe^{3+} .

4. A photostable titanium dioxide sol according to claim 1, wherein said further stabilizing agent is selected from the group consisting of $[\text{F}^{--}, \text{IO}_3^{--}, \text{SO}_4^{2-}, \text{NO}_3^{--}]$ inorganic fluorides, inorganic iodates and inorganic nitrates.

5. A photostable titanium dioxide sol according to claim 1, wherein said further stabilizing agent is EDTA.

6. A photostable titanium dioxide sol according to claim 1, wherein said further stabilizing agent is a perchlorate or a peroxodisulfate.

7. A photostable titanium dioxide sol according to claim 1, wherein said hydroxycarboxylic acid is citric acid or tartaric acid; said complexing agent is EDTA; and said oxidizing agents are perchlorates and peroxodisulfates.

8. A titanium dioxide sol according to claim 1, wherein said oxidizing agents are hydrogen peroxide, perchlorates, perborates and peroxodisulfates.

9. A process for the preparation of a photostable titanium dioxide sol, said sol comprising titanium dioxide sol particles, and an aqueous dispersion medium, said process comprising:

stabilizing said sol by adding 5-40 mole %, based on TiO_2 content of said sol, of a hydroxycarboxylic acid or a salt, ester or amide thereof as a stabilizing

agent, and

after stabilization of said sol with said hydroxycarboxylic acid or a salt, ester or amide thereof, said sol having a pH of 6-8, adding 0.1-20 mole %, based on TiO_2 content of said sol, of a further stabilizing agent selected from the group consisting of metal ions, anions, one or more complexing agents, one or more oxidizing agents, and combinations thereof.

10. A process for preparing a dried photostable titanium dioxide sol, comprising:

drying a sol prepared according to claim 9, and further comprising drying said sol.

12. A process for precipitating a photostable titanium dioxide sol comprising: adding an alcohol to a sol prepared according to claim 9.

13. In a cosmetic formulation containing a carrier and a titanium dioxide sol particle, the improvement wherein said formulation contains a titanium dioxide sol prepared according to claim 9.

17. A titanium dioxide sol comprising:

titanium dioxide sol particles having a particle size of 5-200 nm,

an aqueous dispersion medium,

5-40 mole %, based on TiO_2 content of sol, of a stabilizing agent selected from the group consisting of hydroxy carboxylic acid having 1-6 hydroxy groups and 1-4 --COOH groups, a monoalkyl amide thereof wherein the alkyl group has 1-10 C atoms, a dialkyl amide thereof wherein each, alkyl group has 1-10 C atoms, an alkali metal salt thereof, an alkaline earth salt thereof, and an amine salt thereof, and

0.1-20 mole %, based on TiO_2 content of sol, of a further stabilizing agent, wherein said further stabilizing agent is at least one metal ion, at least one anion, at least one complexing agent, at least one oxidizing agent, or combinations thereof,

wherein said at least one metal ion is La^{2+} , Zn^{2+} , Sn^{4+} , Mn^{2+} , Ce^{3+} , Fe^{2+} , Fe^{3+} , or combinations thereof,

wherein said at least one anion are an inorganic fluoride, an inorganic iodate, an inorganic nitrate or combinations thereof,

wherein said at least one complexing agent is EDTA, nitrilotriacetic acid, 1,2-di-aminocyclohexylene-dinitrilotetraacetic acid, diethylenetriaminepentaacetic acid, N-(2-hydroxy-ethyl)-ethylenediamino-N,N,N-triacetic acid, tetraethylenetetraaminohexaacetic acid or combinations thereof, and

wherein said at least one oxidizing agent is an inorganic peroxide or combinations thereof.

18. A titanium dioxide sol according to claim 17, wherein said at least one oxidizing agent is hydrogen peroxide, a perchlorate, a perborate, a peroxodisulfate or combinations thereof.

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L5: Entry 10 of 45

File: USPT

Mar 30, 1999

US-PAT-NO: 5888484

DOCUMENT-IDENTIFIER: US 5888484 A

TITLE: Composition for bleaching hair

DATE-ISSUED: March 30, 1999

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Schmitt; Manfred	Heppenheim			DE
Gottmann; Holger	Brensbach-Wersau			DE
Balzer; Wolfgang R.	Alsbach			DE
Schiemann; Hartmut	Hunfeld			DE

US-CL-CURRENT: 424/62; 132/208, 424/613, 424/70.13, 424/70.16, 424/DIG.3

CLAIMS:

We claim:

1. A storage-stable, paste-like bleaching agent suspension from which a bleaching composition is made by mixing the bleaching agent suspension with an oxidizing agent, said bleaching agent suspension containing less than 2.5% by weight water and comprising a mixture of at least one inorganic persalt, at least one alkaline reacting salt, from 0.5 to 20 percent by weight of a thickening substance and from 26.5 to 35 percent by weight of at least one oil or wax;

wherein said thickening substance consists essentially of an acrylic acid polymer and at least one polymer selected from the group consisting of celluloses, alginates and polysaccharides, wherein said acrylic acid polymer is present in an amount of from 0.1 to 3 percent by weight.

2. The bleaching agent suspension as defined in claim 1, containing no surfactant.

3. The bleaching agent suspension as defined in claim 1, containing no surfactant and no water.

4. The bleaching agent suspension as defined in claim 1, wherein said at least one inorganic persalt is at least one member selected from the group consisting of sodium persulfate, potassium persulfate and ammonium persulfate.

5. The bleaching agent suspension as defined in claim 1, containing from 30 to 65 percent by weight of said at least one inorganic persalt.

6. The bleaching agent suspension as defined in claim 1, wherein said at least one alkaline reacting salt is at least one member selected from the group consisting of sodium carbonate, sodium hydrogen carbonate, magnesium carbonate, ammonium carbonate, ammonium hydrogen carbonate and sodium silicate.

7. The bleaching agent suspension as defined in claim 1, containing from 15 to 45 percent by weight of said at least one alkaline reacting salt.

8. The bleaching agent suspension as defined in claim 1, wherein said at least one polymer in the thickening substance is selected from the group consisting of methylcelluloses, ethylcelluloses, hydroxyethylcelluloses, methylhydroxyethylcelluloses, methylhydroxypropylcelluloses, carboxymethylcelluloses, alginic acid, sodium alginate, ammonium alginate, calcium alginate, gum arabic, guar gum and xanthan gum.

9. The bleaching agent suspension as defined in claim 1, wherein said at least one oil or wax is at least one member selected from the group consisting of isopropylpalmitate, octylpalmitate, isocetylpalmitate and bees wax.

10. The bleaching agent suspension as defined in claim 1, further comprising at least one auxiliary cosmetic additive ingredient.

11. The bleaching agent suspension as defined in claim 1, further comprising at least one auxiliary cosmetic additive ingredient selected from the group consisting of silicon dioxide, titanium dioxide, ethylenediaminetetraacetic acid, dye compounds and perfumes.

12. A storage-stable, paste-like bleaching agent suspension from which a bleaching composition is made by mixing the suspension with an oxidizing agent, said bleaching agent suspension containing less than 2.5 percent by weight water and comprising a mixture of 30 to 65 percent by weight of at least one inorganic persalt, from 15 to 45 percent by weight of at least one alkaline reacting salt, from 0.5 to 20 percent by weight of a thickening substance and from 26.5 to 35 percent by weight of at least one oil or wax, wherein said thickening substance consists essentially of an acrylic acid polymer and at least one polymer selected from the group consisting of celluloses, alginates and polysaccharides, wherein said acrylic acid polymer is present in an amount of from 0.1 to 3 percent by weight.

13. A two-component bleaching composition product consisting of

a storage-stable, paste-like bleaching agent suspension from which a bleaching composition is made by mixing the bleaching agent suspension with an oxidizing agent,

wherein said bleaching agent suspension contains less than 2.5% by weight water and comprises a mixture of at least one inorganic persalt, at least one alkaline reacting salt, from 0.5 to 20 percent by weight of a thickening substance and from 26.5 to 35 percent by weight of at least one oil or wax, wherein said thickening substance consists essentially of an acrylic acid polymer and at least one polymer selected from the group consisting of celluloses, alginates and polysaccharides, wherein said acrylic acid polymer is present in an amount of from 0.1 to 3 percent by weight; and

the oxidizing agent, wherein the oxidizing agent consists of an aqueous emulsion or solution containing from 6 to 12 percent hydrogen peroxide, and

wherein said bleaching agent suspension is packaged separately from said aqueous emulsion or solution.

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File: USPT

Mar 30, 1999

DOCUMENT-IDENTIFIER: US 5888484 A

TITLE: Composition for bleaching hair

CLAIMS:

4. The bleaching agent suspension as defined in claim 1, wherein said at least one inorganic persalt is at least one member selected from the group consisting of sodium persulfate, potassium persulfate and ammonium persulfate.

11. The bleaching agent suspension as defined in claim 1, further comprising at least one auxiliary cosmetic additive ingredient selected from the group consisting of silicon dioxide, titanium dioxide, ethylenediaminetetraacetic acid, dye compounds and perfumes.

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L7: Entry 3 of 106

File: USPT

Sep 10, 2002

DOCUMENT-IDENTIFIER: US 6447563 B1

TITLE: Chemical mechanical polishing slurry system having an activator solution

Brief Summary Text (7):

In the case of CMP of metals, the chemical action is generally considered to take one of two forms. In the first mechanism, the chemicals in the solution react with the metal layer to continuously form an oxide layer on the surface of the metal. This generally requires the addition of an oxidizer to the solution such as hydrogen peroxide, ferric nitrate, etc. Then the mechanical abrasive action of the particles continuously and simultaneously removes this oxide layer. A judicious balance of these two processes obtains optimum results in terms of removal rate and polished surface quality.

Brief Summary Text (10):

CMP metal slurries, on the other hand, are two part mixtures consisting of a dispersion and an oxidizer. The dispersion comprises an abrasive, an acid to lower the pH to about 2 to 6, optionally a surfactant which maintains the abrasive in suspension and other chemicals tailored to the metal being polished. An example is a tungsten layer slurry called Biplanar.RTM. made by EKC. The dispersion is an acidic dispersion (approximately pH of 3, with 5 to 15% alumina particles). Acids reportedly used in the slurry include carboxylic acids or nitric acid. At the point of use, the dispersion is mixed with an oxidizer, such as hydrogen peroxide or ferric nitrate, to form the slurry that will be used to polish the metal layers.

Brief Summary Text (27):

The novel activator comprises chemicals that are customized to the particular metal being polished. For example, the activator of the present invention may include an oxidizer. Any suitable oxidizer may be used. Examples of a suitable oxidizer are hydrogen peroxide, potassium ferricyanide, potassium dichromate, potassium iodate, potassium bromate, vanadium trioxide, hypochlorous acid, sodium hypochlorite, potassium hypochlorite, calcium hypochlorite, magnesium hypochlorite, ferric nitrate, KMgO.sub.4 , and mixtures thereof. The preferred oxidizer is hydrogen peroxide. The amount of oxidizer is typically 0.01% to 10%, and preferably 0.1 to 5% by weight of the total weight of the slurry.

Brief Summary Text (35):

In a preferred embodiment, the activator solution comprises: (a) about 0.05 wt. % to 5 wt. % of hydrogen peroxide; (b) about 0.05 wt. % to 3 wt. % of propanoic acid; (c) about 0.02 wt. % to 1.5 wt. % of benzotriazole; and (d) about 0.01 wt. % to 2 wt. % of ethanolamine.

Detailed Description Text (4):

A copper slurry was prepared by utilizing a silica dispersion containing 12% fumed silica. The silica dispersion was mixed with an activator containing hydrogen peroxide and propanoic acid. The final copper slurry mixture contained 1% H.sub.2 O.sub.2 by weight, 4% fumed silica by weight and 0.1 molar propanoic acid. Copper wafers were obtained by sputter deposition on a silicon wafer and were polished using the copper slurry with a Rodel IC1400 pad and IPEC 472 tool. The removal rates were in excess of 450 nm, the non-uniformity was less than 5% and the selectivity of copper to SiO_2 was over 200. The passive etch rate without mechanical polishing was 10 nm/minute. In comparison, a commercial copper slurry (Rodel QC 1020) had similar performance.

CLAIMS:

2. The system of claim 1 wherein said abrasive is selected from the group consisting of: silica, alumina, silicon carbide, silicon nitride, iron oxide, ceria, zirconium oxide, tin oxide, titanium dioxide and mixtures thereof.
20. The activator of claim 14 wherein said metal layer is tungsten, said oxidizer is selected from the group consisting of: hydrogen peroxide and ferric nitrate, and said acid is selected from the group consisting of: lactic acid and nitric acid.
22. The activator of claim 14 wherein said metal layer is copper and wherein said activator comprises hydrogen peroxide; an acid selected from the group consisting of propanoic acid, phthalic acid and citric acid; and benzotriazole.
23. The activator of claim 14 wherein said metal layer is tantalum and wherein said activator comprises hydrogen peroxide, ammonium fluoride and at least one other component.
24. The activator of claim 14 wherein said metal layer is aluminum and wherein said activator comprises hydrogen peroxide, ammonium fluoride and a weak acid.
25. The activator of claim 14 wherein said metal layer is titanium and wherein said activator comprises hydrogen peroxide, ammonium fluoride and a weak acid.
26. An activator solution, free of abrasives, for a slurry system used for polishing metal layers, said slurry system having a first part and a second part, said activator solution comprising: (a) about 0.05 wt. % to 5 wt. % of hydrogen peroxide; (b) about 0.05 wt. % to 3 wt. % of propanoic acid; (c) about 0.02 wt. % to 1.5 wt. % of benzotriazole; and (d) about 0.01 wt. % to 2 wt. % of ethanolamine; wherein said first part is a dispersion solution and said activator solution is said second part of said slurry system, and said activator solution is packaged separately from said dispersion solution.
28. The process of claim 27 wherein said abrasive is selected from the group consisting of: silica, alumina, silicon carbide, silicon nitride, iron oxide, ceria, zirconium oxide, tin oxide, titanium dioxide and mixtures thereof.

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L7: Entry 3 of 106

File: USPT

Sep 10, 2002

US-PAT-NO: 6447563

DOCUMENT-IDENTIFIER: US 6447563 B1

TITLE: Chemical mechanical polishing slurry system having an activator solution

DATE-ISSUED: September 10, 2002

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Mahulikar; Deepak	Madison	CT		

ASSIGNEE-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY	TYPE CODE
Arch Specialty Chemicals, Inc.	Norwalk	CT			02

APPL-NO: 09/ 425358 [PALM]

DATE FILED: October 22, 1999

PARENT-CASE:

This application claims priority from U.S. Provisional Application Ser. No. 60/105,366 filed on Oct. 23, 1998.

INT-CL: [07] C09 K 3/14, C09 G 1/02, B24 B 1/00

US-CL-ISSUED: 51/309; 51/307, 51/308, 106/3, 438/692, 438/693, 252/79.2, 252/79.3, 252/79.4

US-CL-CURRENT: 51/309; 106/3, 252/79.2, 252/79.3, 252/79.4, 438/692, 438/693, 51/307, 51/308

FIELD-OF-SEARCH: 51/307, 51/308, 51/309, 106/3, 438/692, 438/693, 510/167, 510/175, 510/397, 252/79.2, 252/79.3, 252/79.4, 252/79.5, 216/96, 216/89, 216/102, 216/103, 216/104, 216/105, 216/106, 216/107, 216/108, 216/109

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

Search Selected

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	PAT-NO	ISSUE-DATE	PATENTEE-NAME	US-CL
<input type="checkbox"/>	<u>4959113</u>	September 1990	Roberts	
<input type="checkbox"/>	<u>5013871</u>	May 1991	Mahulikar et al.	
<input type="checkbox"/>	<u>5084071</u>	January 1992	Nenadic et al.	
<input type="checkbox"/>	<u>5209816</u>	May 1993	Yu et al.	
<input type="checkbox"/>	<u>5230833</u>	July 1993	Romberger et al.	
<input type="checkbox"/>	<u>5264010</u>	November 1993	Brancaleoni et al.	
<input type="checkbox"/>	<u>5266088</u>	November 1993	Sandusky et al.	51/309
<input type="checkbox"/>	<u>5318927</u>	June 1994	Sandhu et al.	
<input type="checkbox"/>	<u>5340370</u>	August 1994	Cadien et al.	
<input type="checkbox"/>	<u>5354490</u>	October 1994	Yu et al.	
<input type="checkbox"/>	<u>5366542</u>	November 1994	Yamada et al.	51/309
<input type="checkbox"/>	<u>5476606</u>	December 1995	Brancaleoni et al.	
<input type="checkbox"/>	<u>5527423</u>	June 1996	Neville et al.	51/308
<input type="checkbox"/>	<u>5700383</u>	December 1997	Feller et al.	51/308
<input type="checkbox"/>	<u>5783489</u>	July 1998	Kaufman et al.	51/309
<input type="checkbox"/>	<u>5800577</u>	September 1998	Kido	51/308
<input type="checkbox"/>	<u>5866031</u>	February 1999	Carpio et al.	252/79.2
<input type="checkbox"/>	<u>5954997</u>	September 1999	Kaufman et al.	252/79.2
<input type="checkbox"/>	<u>5993686</u>	November 1999	Streinz et al.	51/309
<input type="checkbox"/>	<u>6063306</u>	May 2000	Kaufman et al.	252/79.2
<input type="checkbox"/>	<u>6083840</u>	July 2000	Mravic et al.	252/79.4
<input type="checkbox"/>	<u>6143059</u>	November 2000	Tangi et al.	106/1.22

FOREIGN PATENT DOCUMENTS

FOREIGN-PAT-NO	PUBN-DATE	COUNTRY	US-CL
WO 98/04646	February 1998	WO	

ART-UNIT: 1755

PRIMARY-EXAMINER: Marcheschi; Michael

ABSTRACT:

This invention relates to a CMP slurry system for use in semiconductor manufacturing. The slurry system comprises two parts. The first part is a generic dispersion that only contains an abrasive and, optionally, a surfactant and a stabilizing agent. The generic dispersion can be used for polishing metals as well as interlayer dielectrics (ILD). The second part is a novel activator solution comprising at least two components selected from the group consisting of: an oxidizer, acids, amines, chelating agents, fluorine-containing compounds, corrosion inhibitors, buffering agents, surfactants, biological agents and mixtures thereof.

39 Claims, 0 Drawing figures

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L7: Entry 6 of 106

File: USPT

Jul 16, 2002

DOCUMENT-IDENTIFIER: US 6420437 B1

TITLE: Titanium oxide colloidal sol and process for the preparation thereof

Brief Summary Text (11):

As a means for solving the above-mentioned problems, Japanese Unexamined Patent Publication No. 9-71,418 discloses a sol liquid comprising hydrogen peroxide and titanium dioxide and a method of producing the sol liquid. The sol liquid is advantageous in that the sol liquid can be neutralized. However, since an oxidizing agent is contained, the sol liquid is disadvantageous in that the effect of the sol liquid for preventing the metal corrosion is low, the sol liquid is colored yellow, and a colorless coating is difficult to be formed from the sol liquid unless the coating is heat-dried.

Detailed Description Text (31):

As explained in detail above, the titanium dioxide colloid sol of the present invention is a neutral photocatalytic titanium dioxide colloid sol having high stability and dispersibility and usable as a coating material for articles, for example, metal articles having a low resistance to corrosion and organic articles having a low resistance to deterioration. When the titanium dioxide colloid sol is employed for the purpose of decomposition of stains, ultraviolet ray absorption, sterilization, gas decomposition, and water treatment, the sol can be applied to wide various materials, and the problems of the working atmosphere and safety can be solved. Therefore, the titanium dioxide colloid sol and the method of producing the sol of the present invention are industrially valuable.

CLAIMS:

1. A titanium dioxide colloid sol comprising 50 to 100 parts by weight of titanium dioxide colloidal particles charged with negative electricity and 5 to 50 parts by weight of a complexing agent and 1 to 50 parts by weight of an alkaline substance, the complexing agent comprising at least one member selected from the group consisting of condensed phosphoric acids and condensed phosphate salts.
2. The titanium dioxide colloid sol as claimed in claim 1, wherein the pH value of the titanium dioxide sol is 5 to 10.
3. The titanium dioxide colloid sol as claimed in claim 1, wherein the alkaline substance comprises at least one member selected from the group consisting of ammonium compounds, alkali metal compounds and amine compounds.
4. The titanium dioxide colloid sol as claimed in claim 1, wherein the alkaline substance comprises at least one member selected from the group consisting of oxazine, piperidine and choline compounds.
5. A method of producing a titanium dioxide colloid sol characterized by mixing an alkaline substance into an acid titanium dioxide colloid sol containing 50 to 100 parts by weight of titanium dioxide colloid sol and 5 to 50 parts by weight of a complexing agent, to adjust the pH value of the sol to a level of 5 to 10, and to thereby cause the titanium dioxide colloidal particles to be charged with negative electricity, the complexing agent comprising at least one member selected from the group consisting of condensed phosphoric acid and condensed phosphate salts.
6. The method of producing the titanium dioxide colloid sol as claimed in claim 5,

wherein the alkaline substance comprises at least one member selected from the group consisting of ammonium compounds, alkali metal compounds and amine compounds.

7. The method of producing the titanium dioxide colloid sol as claimed in claim 5, wherein the alkaline substance comprises at least one member selected from the group consisting of oxazine, piperidine and choline compounds.

8. A method of producing al titanium dioxide colloid sol, characterized by mixing an alkaline substance into an acid titanium dioxide colloid sol comprising 50 to 100 parts by weight of titanium dioxide colloidal particles and 5 to 50 parts by weight of a complexing agent to adjust the pH value of the resultant sol to a level of 6 to 12, and applying a deionization treatment to the sol to thereby cause the titanium dioxide colloidal particles to be charged with negative electricity.

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L7: Entry 6 of 106

File: USPT

Jul 16, 2002

US-PAT-NO: 6420437

DOCUMENT-IDENTIFIER: US 6420437 B1

TITLE: Titanium oxide colloidal sol and process for the preparation thereof

DATE-ISSUED: July 16, 2002

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Mori; Kazuhiko	Tokyo			JP
Nakamura; Mitsuru	Tokyo			JP
Tanaka; Masanobu	Tokyo			JP

US-CL-CURRENT: 516/90; 106/13, 106/287.19, 106/440, 106/448, 252/588, 502/350

CLAIMS:

What the claimed is:

1. A titanium dioxide colloid sol comprising 50 to 100 parts by weight of titanium dioxide colloidal particles charged with negative electricity and 5 to 50 parts by weight of a complexing agent and 1 to 50 parts by weight of an alkaline substance, the complexing agent comprising at least one member selected from the group consisting of condensed phosphoric acids and condensed phosphate salts.
2. The titanium dioxide colloid sol as claimed in claim 1, wherein the pH value of the titanium dioxide sol is 5 to 10.
3. The titanium dioxide colloid sol as claimed in claim 1, wherein the alkaline substance comprises at least one member selected from the group consisting of ammonium compounds, alkali metal compounds and amine compounds.
4. The titanium dioxide colloid sol as claimed in claim 1, wherein the alkaline substance comprises at least one member selected from the group consisting of oxazine, piperidine and choline compounds.
5. A method of producing a titanium dioxide colloid sol characterized by mixing an alkaline substance into an acid titanium dioxide colloid sol containing 50 to 100 parts by weight of titanium dioxide colloid sol and 5 to 50 parts by weight of a complexing agent, to adjust the pH value of the sol to a level of 5 to 10, and to thereby cause the titanium dioxide colloidal particles to be charged with negative electricity, the complexing agent comprising at least one member selected from the group consisting of condensed phosphoric acid and condensed phosphate salts.
6. The method of producing the titanium dioxide colloid sol as claimed in claim 5, wherein the alkaline substance comprises at least one member selected from the group consisting of ammonium compounds, alkali metal compounds and amine compounds.
7. The method of producing the titanium dioxide colloid sol as claimed in claim 5, wherein the alkaline substance comprises at least one member selected from the group consisting of oxazine, piperidine and choline compounds.

8. A method of producing al titanium dioxide colloid sol, characterized by mixing an alkaline substance into an acid titanium dioxide colloid sol comprising 50 to 100 parts by weight of titanium dioxide colloidal particles and 5 to 50 parts by weight of a complexing agent to adjust the pH value of the resultant sol to a level of 6 to 12, and applying a deionization treatment to the sol to thereby cause the titanium dioxide colloidal particles to be charged with negative electricity.

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L7: Entry 6 of 106

File: USPT

Jul 16, 2002

US-PAT-NO: 6420437

DOCUMENT-IDENTIFIER: US 6420437 B1

TITLE: Titanium oxide colloidal sol and process for the preparation thereof

DATE-ISSUED: July 16, 2002

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Mori; Kazuhiko	Tokyo			JP
Nakamura; Mitsuru	Tokyo			JP
Tanaka; Masanobu	Tokyo			JP

ASSIGNEE-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY	TYPE CODE
Nihon Parkerizing Co., Ltd.	Tokyo			JP	03

APPL-NO: 09/ 601034 [PALM]

DATE FILED: July 26, 2000

FOREIGN-APPL-PRIORITY-DATA:

COUNTRY	APPL-NO	APPL-DATE
JP	10-013811	January 27, 1998
JP	10-282938	October 5, 1998

PCT-DATA:

APPL-NO	DATE-FILED	PUB-NO	PUB-DATE	371-DATE	102(E)-DATE
PCT/JP99/00342	January 27, 1999	WO99/37582	Jul 29, 1999	Jul 26, 2000	Jul 26, 2000

INT-CL: [07] B01 F 17/14, B01 F 17/16, C09 C 1/36, C09 K 3/18, F21 V 9/06

US-CL-ISSUED: 516/90; 106/13, 106/287.19, 106/440, 106/448, 252/588, 502/350

US-CL-CURRENT: 516/90; 106/13, 106/287.19, 106/440, 106/448, 252/588, 502/350

FIELD-OF-SEARCH: 516/90, 106/287.19, 106/440, 106/448, 106/13, 502/350, 252/588

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

Search Selected

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	PAT-NO	ISSUE-DATE	PATENTEE-NAME	US-CL
<input type="checkbox"/>	<u>2819177</u>	January 1958	Conn et al.	
<input type="checkbox"/>	<u>3147131</u>	September 1964	Kingsbury	106/448
<input type="checkbox"/>	<u>3567479</u>	March 1971	Portes et al.	106/448
<input type="checkbox"/>	<u>3582275</u>	June 1971	Sugahara et al.	
<input type="checkbox"/>	<u>3663284</u>	May 1972	Stancioff et al.	106/448 X
<input type="checkbox"/>	<u>4448609</u>	May 1984	Tear et al.	516/90 X
<input type="checkbox"/>	<u>4576921</u>	March 1986	Lane	516/90 X
<input type="checkbox"/>	<u>5049309</u>	September 1991	Sakamoto et al.	516/90
<input type="checkbox"/>	<u>5059248</u>	October 1991	Signorino et al.	516/90 X
<input type="checkbox"/>	<u>5698205</u>	December 1997	Bruckner et al.	516/90 X
<input type="checkbox"/>	<u>5759251</u>	June 1998	Nakamura et al.	106/287.19 X

FOREIGN PATENT DOCUMENTS

FOREIGN-PAT-NO	PUBN-DATE	COUNTRY	US-CL
1 271 862	July 1968	DE	106/448
0376216	July 1990	EP	
0684075	November 1995	EP	
7-89722	April 1995	JP	
10-18082	January 1998	JP	

OTHER PUBLICATIONS

Riskin et al. "Colloidal stability of titanium dioxide sols in concentrated hydrochloric Acid solutions" (1970), Chemical Abstracts No. 79:70516.

ART-UNIT: 1712

PRIMARY-EXAMINER: Lovering; Richard D.

ABSTRACT:

A neutral titanium dioxide colloid sol having a high stability in a neutral range and capable of forming a colorless transparent coating even when dried at room temperature is produced by mixing an acid titanium dioxide sol comprising 50 to 100 parts by weight of titanium dioxide colloidal particles and 5 to 50 parts by weight of a chelating agent for titanium ions with 1 to 50 parts by weight of an alkaline substance comprising at least one of alkali metal compounds and amine compounds, and optionally by adjusting the pH value of the liquid mixture to 5 to 10, or adjusting the pH value of the liquid mixture to 6 to 10, and then applying a deionization treatment to the mixture to thereby charge the titanium dioxide colloidal particles with negative electricity.

8 Claims, 0 Drawing figures

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L7: Entry 10 of 106

File: USPT

Jun 26, 2001

US-PAT-NO: 6251150

DOCUMENT-IDENTIFIER: US 6251150 B1

TITLE: Slurry composition and method of chemical mechanical polishing using same

DATE-ISSUED: June 26, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Small; Robert James	Dublin	CA		
Peterson; Maria Louise	Menlo Park	CA		
Troung; Tuan	San Jose	CA		
Bonneau; Lionel	Cran Gevrier			FR
Drouget; Jean Claude	Annecy le Vieux			FR

US-CL-CURRENT: 51/307; 106/3, 423/593, 423/594, 423/595, 423/598, 423/599, 423/600,
438/692, 438/693, 51/309, 510/397

CLAIMS:

That which is claimed:

1. A slurry composition for chemical mechanical polishing comprising spinel particles having the formula $\text{AO} \cdot \text{xZ} \cdot \text{O}_3$ wherein A is at least one divalent cation, Z is at least one trivalent cation, and $0.01 \leq \text{x} \leq 100$, said particles having a mean crystallite diameter of between about 5 and about 500 nm and a mean aggregate particle diameter of between about 20 and about 5000 nm.
2. The slurry composition according to claim 1 being substantially free of particles having a Mohs hardness of greater than 8.5.
3. The slurry composition according to claim 1 being substantially free of alpha phase alumina particles.
4. The slurry composition according to claim 1, wherein said particles have a specific area of between about 5 m^2/g and about 200 m^2/g .
5. The slurry composition according to claim 1, wherein $1 \leq \text{x} \leq 100$.
6. The slurry composition according to claim 1, wherein $1 \leq \text{x} \leq 12.5$.
7. The slurry composition according to claim 1, wherein A is selected from the group consisting of Mg, Fe^{2+} , Zn, Mn, Ni, Ca, and combinations thereof; and Z is selected from the group consisting of Al, Fe^{3+} , Cr, Ti, and mixtures thereof.
8. The slurry composition according to claim 1, wherein the spinel particles include a small amount of at least one cation selected from the group consisting of Zr, Si, Ga, Cu, Co, V, B, Li, rare earth cations, and mixtures thereof.
9. The slurry composition according to claim 1, wherein A is Mg and Z is Al.
10. The slurry composition according to claim 1, wherein A is Zn and Z is Al.

11. The slurry composition according to claim 1 further comprising an oxidizing agent.
12. The slurry composition according to claim 11, wherein the oxidizing agent is ferric nitrate.
13. The slurry composition according to claim 11, wherein the oxidizing agent is periodic acid.
14. The slurry composition according to claim 11, wherein the oxidizing agent is hydrogen peroxide.
15. The slurry composition according to claim 11, wherein the oxidizing agent is selected from the group consisting of hydroxylamine and hydroxylamine salts.
16. The slurry composition according to claim 1, wherein the spinel particles are present in an amount between about 0.1 and about 10 percent by weight.
17. The slurry composition according to claim 1, further comprising additional particles selected from the group consisting of alumina, silica, ceria, germania, zirconia, tin oxide, silicon carbide, titanium dioxide, titanium carbide, and mixtures thereof, said additional particles present in an amount between about 0.1 and about 10 percent by weight.
18. The slurry composition according to claim 1 having a Zeta potential of greater than about +10 mV or less than about -10 mV.
19. A slurry composition for chemical mechanical polishing comprising: 0.1 to 10% by weight of spinel particles having the formula $AO \cdot xZ \cdot 2O \cdot 3$ wherein A is at least one divalent cation, Z is at least one trivalent cation, and $1 \leq x \leq 12$, said particles having a mean crystallite diameter of between about 5 and about 500 nm and a mean aggregate particle diameter of between about 20 and about 5000 nm, the slurry composition being substantially free of particles having a Mohs hardness of greater than 8.5;
1 to 15% of an oxidizing agent; and
water.
20. A slurry composition for chemical mechanical polishing comprising between 0.1 and 1 percent by weight of spinel particles having the formula $MgO \cdot xAl \cdot 2O \cdot 3$ wherein $1 \leq x \leq 12$, a specific area of between about 5 m²/g and about 200 m²/g, a mean crystallite diameter of between about 5 and about 500 nm, and a mean aggregate particle diameter of between about 20 and about 5000 nm, said slurry composition being substantially free of alpha phase alumina particles.
21. A slurry composition for chemical mechanical polishing comprising between 0.1 and 1 percent by weight of spinel particles having the formula $ZnO \cdot xAl \cdot 2O \cdot 3$ wherein $1 \leq x \leq 12$, a specific area of between about 5 m²/g and about 200 m²/g, a mean crystallite diameter of between about 5 and about 500 nm, and a mean aggregate particle diameter of between about 20 and about 5000 nm, said slurry composition being substantially free of alpha phase alumina particles.
22. A slurry composition for chemical mechanical polishing comprising spinel particles having the formula $MgO \cdot xAl \cdot 2O \cdot 3$, said particles having a mean crystallite diameter of between about 5 and about 500 nm and a mean aggregate particle diameter of between about 20 and about 5000 nm, wherein the ratio of $MgO:Al \cdot 2O \cdot 3$ (1/x) and the specific area are selected to be in the region above the SN6(-), SN3(-), and SN1(-) tie line as defined in FIG. 1.
23. A method of chemical mechanical polishing a substrate comprising the steps of:

applying a slurry composition to a surface of a substrate, said slurry composition comprising spinel particles having the formula $A_0 \cdot xZ \cdot 2O \cdot 3$ wherein A is at least one divalent cation, Z is at least one trivalent cation, $0.01 \leq x \leq 100$, said particles having a mean crystallite diameter of between about 5 and about 500 nm and a mean aggregate particle diameter of between about 20 and about 5000 nm; and

polishing the surface of the substrate.

24. The method according to claim 23, wherein said applying step comprises applying a slurry composition that is substantially free of particles having a Mohs hardness of greater than 8.5.

25. The method according to claim 23, wherein said applying step comprises applying a slurry composition that is substantially free of alpha phase alumina particles.

26. The method according to claim 23, wherein said applying step comprises applying a slurry composition comprising spinel particles having a specific area between about 5 m²/g and about 200 m²/g.

27. The method according to claim 23, wherein said applying step comprises applying a slurry composition wherein $1 \leq x \leq 100$.

28. The method according to claim 23, wherein said applying step comprises applying a slurry composition wherein $1 \leq x \leq 12.5$.

29. The method according to claim 23, wherein said applying step comprises applying a slurry composition wherein A is selected from the group consisting of Mg, Fe²⁺, Zn, Mn, Ni, Ca, and combinations thereof; and Z is selected from the group consisting of Al, Fe³⁺, Cr, Ti, and mixtures thereof.

30. The method according to claim 23, wherein said applying step comprises applying a slurry composition wherein the spinel particles include a small amount of at least one cation selected from the group consisting of Zr, Si, Ga, Cu, Co, V, B, Li, rare earth cations, and mixtures thereof.

31. The method according to claim 23, wherein said applying step comprises applying a slurry composition wherein A is Mg and Z is Al.

32. The method according to claim 23, wherein said applying step comprises applying a slurry composition wherein A is Zn and Z is Al.

33. The method according to claim 23, wherein said applying step comprises applying a slurry composition further comprising additional particles selected from the group consisting of alumina, silica, ceria, germania, zirconia, tin oxide, silicon carbide, titanium dioxide, titanium carbide, and mixtures thereof, said additional particles present in an amount between about 0.1 and about 10 percent by weight.

34. The method according to claim 23, wherein said applying step comprises applying a slurry composition having a Zeta potential of greater than about +10 mV or less than about -10 mV.

35. The method according to claim 23, further comprising the step of preparing spinel particles by thermally decomposing one or more source compounds comprising A cations and Z cations.

36. The method according to claim 35, wherein said preparing step comprises preparing spinel particles wherein Z is Al by decomposing ammonium alum at an elevated temperature with at least one source compound including A cations.

37. The method according to claim 23, wherein the surface is a metal or metal oxide surface.

38. The method according to claim 23, wherein the surface is selected from the group consisting of tungsten, aluminum, copper, titanium, titanium nitride, tantalum, silicon nitride, low-K dielectric, high-K dielectric, silicon, polysilicon, and tetraethoxysilane surfaces.

39. The method according to claim 23, wherein the substrate is selected from the group consisting of semiconductor wafers, metals, ceramics, hat disk surfaces, and magnetic rewrite disk heads.

40. A substrate polished according to the method of claim 23.

41. The slurry composition according to claim 1, wherein said particles have a specific area of between 50 m.²/g and 150 m.²/g.

42. The slurry composition according to claim 1, wherein said particles have a crystallite phase distribution including greater than about 95% gamma spinel phase particles.

43. A slurry composition comprising spinel particles having the formula $A_0.xZ_{0.3}O$ wherein A is at least one divalent cation, Z is at least one trivalent cation, and $0.01 \leq x \leq 100$, said particles having a mean crystallite diameter of between about 5 and about 500 nm and a mean aggregate particle diameter of between about 20 and about 5000 nm, said slurry composition being suitable for chemical mechanical polishing.

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L7: Entry 10 of 106

File: USPT

Jun 26, 2001

DOCUMENT-IDENTIFIER: US 6251150 B1

TITLE: Slurry composition and method of chemical mechanical polishing using same

Brief Summary Text (16):

In addition, because of chemical stability problems, many slurries must be prepared at the point of use because they have little or no pot life. For example, slurries that include hydrogen peroxide oxidizing agents are generally not stable when premixed with the other slurry components and therefore have to be delivered to the polishing equipment with separate pumping systems and mixed at the point of use. Moreover, other slurries, such as those that use potassium iodate system require special handling. Metal polishing slurries can also contribute to mobile ion contamination, can affect the slurry particle distribution and compositions, and can be toxic to the environment.

Brief Summary Text (33):

The slurry compositions of the invention are preferably aqueous compositions and typically include spinel particles and at least one liquid component (other than water) in the aqueous medium. Preferably, the liquid component is an oxidizing agent such as ferric nitrate, periodic acid, hydrogen peroxide, hydroxylamine or a hydroxylamine salt solution. In the slurry compositions, the spinel particles are preferably present in an amount between about 0.1 and about 10 percent by weight. The slurry compositions can also include between 0.1 and 10 percent by weight of other abrasive particles.

Detailed Description Text (16):

Other oxidizing or reducing agents can also be used in the invention including ammonium persulfate; hydrogen peroxide; peracetic acid; periodic acid; ferric nitrate; ammonium peroxy compounds; ammonium bifluoride; polyelectrolytes; organic acids (e.g. malonic acid, succinic acid, tartaric acid, citric acid, and oxalic acid); peroxymonosulfuric acid; potassium peroxymonosulfate or malonamide; lithium periodate; potassium iodate; potassium periodate; perborates; peroxyhydrates; urea hydrogen peroxide complexes; inorganic halides particularly fluorides; nitrates; hypohalites (e.g. hypochlorites); cyanides; permanganates; and the like. Chelating agents can also be used such as alkyl beta-diketone, alkylene tetraacetic acids, aromatic phenolic aldehydes, bis(hydroxypropyl) hydroxylamine, anisaldehyde, alpha hydroxy isobutyric acid, aromatic dioxygenated compounds, catechol compounds and gallic acid compounds. Other additives can also be used such as surfactants, stabilizers, suspension agents, buffers, lubricants and the like.

Detailed Description Text (42):

As demonstrated above, the $\text{MgO} \cdot \text{Al}_{0.2} \text{O}_{0.3}$ spinel particles in periodic acid produced better oxide erosion results than silica in hydrogen peroxide. In ferric nitrate, the combination of $\text{MgO} \cdot \text{Al}_{0.2} \text{O}_{0.3}$ and SiO_2 offers oxide erosion advantages over 50-50 alumina and the wet milled $\text{MgO} \cdot \text{Al}_{0.2} \text{O}_{0.3}$. This combination does not offer oxide erosion advantages over CR-85 alumina but CR-85 alumina scratches the surface and requires an oxide polishing step to remove microscratches.

CLAIMS:

14. The slurry composition according to claim 11, wherein the oxidizing agent is hydrogen peroxide.

17. The slurry composition according to claim 1, further comprising additional particles selected from the group consisting of alumina, silica, ceria, germania, zirconia, tin oxide, silicon carbide, titanium dioxide, titanium carbide, and mixtures thereof, said additional particles present in an amount between about 0.1 and about 10 percent by weight.

33. The method according to claim 23, wherein said applying step comprises applying a slurry composition further comprising additional particles selected from the group consisting of alumina, silica, ceria, germania, zirconia, tin oxide, silicon carbide, titanium dioxide, titanium carbide, and mixtures thereof, said additional particles present in an amount between about 0.1 and about 10 percent by weight.

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L7: Entry 11 of 106

File: USPT

May 15, 2001

US-PAT-NO: 6231343

DOCUMENT-IDENTIFIER: US 6231343 B1

TITLE: Method for bleaching discolored tooth by titanium dioxide photocatalyst

DATE-ISSUED: May 15, 2001

INVENTOR-INFORMATION:

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US-CL-ISSUED: 433/215; 433/216

US-CL-CURRENT: 433/215; 433/216

FIELD-OF-SEARCH: 433/215, 433/216, 433/229, 433/80

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

	PAT-NO	ISSUE-DATE	PATENTEE-NAME	US-CL
<input type="checkbox"/>	<u>5766574</u>	June 1998	Christina-Beck et al.	424/53
<input type="checkbox"/>	<u>5902568</u>	May 1999	Ryles et al.	424/53
<input type="checkbox"/>	<u>5914305</u>	June 1999	Madison et al.	510/367

ART-UNIT: 372

PRIMARY-EXAMINER: Lucchesi; Nicholas D.

ABSTRACT:

The present invention relates to a method for bleaching a discolored tooth, comprising the steps of applying a solution/paste of titanium dioxide powder and hydrogen peroxide solution onto the surface of discolored teeth, and bleaching the tooth based on the photocatalytic action that is produced by irradiating this area with light; and to a bleaching agent for applying onto the surface of discolored tooth to bleach the discolored tooth based on the photocatalytic action that is produced by irradiating this area with light, the bleaching agent comprising as active ingredients a combination of hydrogen peroxide solution and titanium dioxide producing photocatalytic action when irradiated with light.

16 Claims, 0 Drawing figures

WEST☐

L7: Entry 11 of 106

File: USPT

May 15, 2001

US-PAT-NO: 6231343

DOCUMENT-IDENTIFIER: US 6231343 B1

TITLE: Method for bleaching discolored tooth by titanium dioxide photocatalyst

DATE-ISSUED: May 15, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Ishibashi; Takuro	Nagasaki	817-001		JP
Ishibashi; Kouzo	Nagasaki	817-001		JP
Taoda; Hiroshi	Nagoya-shi			JP
Nonami; Toru	Nagoya-shi			JP

US-CL-CURRENT: 433/215; 433/216

CLAIMS:

What is claimed is:

1. A method for bleaching discolored teeth comprising applying a solution or paste or both comprising a titanium dioxide powder and hydrogen peroxide solution onto the surface of the discolored teeth, and irradiating the discolored teeth with light, thereby bleaching the teeth by resultant photocatalytic action.

2. The method for bleaching discolored teeth according to claim 1, wherein said irradiating light is violet or blue visible light.

3. The method for bleaching discolored teeth according to claim 1, which comprises:

a) cleaning surfaces of the discolored teeth;

b) moisture-proofing said cleaned surfaces; and

c) coating said moisture-proofed surfaces with said solution or paste or both of said titanium dioxide powder and said hydrogen peroxide, and irradiating said coated surfaces with visible light.

4. The method for bleaching discolored teeth according to claim 3, wherein step c) is repeated, whereby fresh paste or solution or both is applied and irradiated every 15 to 20 minutes.

5. The method for bleaching discolored teeth according to claim 4, wherein step c) is repeated 2 or 3 times.

6. The method for bleaching discolored teeth according to claim 5, wherein step c) is repeated 4 or 5 times.

7. The method for bleaching discolored teeth according to claim 1, wherein said solution or past further comprises trace amounts of iron salts.

8. A bleaching composition for bleaching discolored teeth by photocatalytic

action produced by irradiation with light, wherein

said bleaching composition comprises as active ingredients a combination of 6% or less hydrogen peroxide solution and crystalline titanium dioxide photocatalyst, wherein

said photocatalyst produces the photocatalytic action and reacts with the hydrogen peroxide to produce active oxygen when irradiated with light.

9. The bleaching composition according to claim 8, which consists essentially of a solution/paste of titanium dioxide with a particle diameter of about 5 to 60 nm and 3% or less hydrogen peroxide solution.

10. The bleaching composition according to claim 8, which further comprises trace amounts of iron salts.

11. A method for producing a bleaching composition for bleaching discolored teeth by photocatalytic action produced by irradiation with light, which method comprises:

blending 6% or less hydrogen peroxide solution with a crystalline titanium dioxide photocatalyst powder, wherein

said photocatalyst producing the photocatalytic action and reacts with hydrogen peroxide to produce active oxygen when irradiated with light.

12. The method for producing a bleaching composition according to claim 11, wherein 3% or less hydrogen peroxide solution is blended with the crystalline titanium dioxide photocatalyst powder.

13. The method for producing a bleaching agent according to claim 11, wherein the crystalline titanium dioxide photocatalyst powder is anatase-type titanium dioxide.

14. A system for bleaching teeth, comprising a combination of a bleaching composition, materials or device for applying said bleaching composition, irradiating device, and optionally other dental treatment materials, wherein the bleaching composition comprises as active ingredients a combination of 6% or less hydrogen peroxide solution and crystalline titanium dioxide photocatalyst,

said photocatalyst producing the photocatalytic action and reacting with the hydrogen peroxide to produce active oxygen when irradiated with light.

15. The system for bleaching teeth according to claim 14, wherein the radiating device generates visible violet light.

16. The system for bleaching teeth according to claim 15, wherein the radiating device generates LED violet light.

WEST

Generate Collection

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L7: Entry 11 of 106

File: USPT

May 15, 2001

DOCUMENT-IDENTIFIER: US 6231343 B1

TITLE: Method for bleaching discolored tooth by titanium dioxide photocatalystAbstract Text (1):

The present invention relates to a method for bleaching a discolored tooth, comprising the steps of applying a solution/paste of titanium dioxide powder and hydrogen peroxide solution onto the surface of discolored teeth, and bleaching the tooth based on the photocatalytic action that is produced by irradiating this area with light; and to a bleaching agent for applying onto the surface of discolored tooth to bleach the discolored tooth based on the photocatalytic action that is produced by irradiating this area with light, the bleaching agent comprising as active ingredients a combination of hydrogen peroxide solution and titanium dioxide producing photocatalytic action when irradiated with light.

Brief Summary Text (3):

The present invention relates to the utilization of photocatalytic action to bleach and remove colored sediment on (stained or discolored) teeth, and more particularly to a method for bleaching discolored teeth comprising the steps of applying a bleaching agent comprising a specific composition having photocatalytic action on the surface of discolored teeth, and bleaching the teeth based on the photocatalytic action that is produced by irradiating this area with light; a novel bleaching agent comprising a solution/paste of 3% or less aqueous hydrogen peroxide and titanium dioxide producing photocatalytic action when irradiated with light, which is useful for such bleaching; a method for producing such a bleaching agent; and a bleaching system comprising a combination of the aforementioned bleaching agent and the like.

Brief Summary Text (5):

There has been increasing demand in recent years for improvements in the contour, alignment, and integrity of teeth in dental therapy, which may be referred to as cosmetic improvements. Recently there have been more cases of patients seeking dental therapy based on a desire for whiter teeth, which is increasingly regarded by young women as an important element of beauty. The causes of dental discoloration and pigmentation or staining are generally classified into so-called extrinsic factors, such as the sedimentation of colored substances (tobacco, tea, etc.), pigment generating bacteria, the discoloration of repair materials (primarily composite resins), and metal salts (primarily amalgams, silver nitrate, and ammonia silver), and intrinsic factors, such as aging, chemicals or drugs (such as fluorine and tetracyclines), dysmetabolism and hereditary diseases, and dental injuries. The latter intrinsically discolored teeth are indicated for bleaching methods to which dental health system treatment is applied.

Brief Summary Text (6):

Several methods have been proposed in the past as methods for the cosmetic improvement of discolored teeth, among which bleaching may be considered a highly effective method for the preservation of dentine, despite drawbacks such as occasional recurrence, when methods suited to individual cases are selected and properly carried out.

Brief Summary Text (9):

1) Bleaching which features the use of 30% H.sub.2 O.sub.2 as the chemical agent, and the combined use of light and heat on the aqueous hydrogen peroxide.

Brief Summary Text (10):

This is a method in which strips of gauze soaked in 30% H.sub.2 O.sub.2 are placed on the front surface of the teeth and are irradiated for 30 minutes from the left and right by two 500 W photographic lamps. In this method, the lamps are brought as close as possible, and the H.sub.2 O.sub.2 must be replenished about every 5 minutes to keep the gauze from drying.

Brief Summary Text (11):

2) Bleaching which features the use of 30% H.sub.2 O.sub.2 as the chemical agent, and the concurrent use of high frequency current on the aqueous hydrogen peroxide.

Brief Summary Text (12):

This is a method in which strips of gauze soaked in 30% H.sub.2 O.sub.2 are placed on the front surface of the teeth, and high frequency current is delivered for 1 second with the spoon-shaped tip of a high frequency scalpel. These operations are repeated 6 to 8 times every 8 seconds, during which time the H.sub.2 O.sub.2 in the gauze must be replenished to prevent it from drying.

Brief Summary Text (13):

3) Bleaching in which a paste comprising Aerosil (silica fine powder) mixed with 35% H.sub.2 O.sub.2 is used as the chemical agent, and the aqueous hydrogen peroxide and Aerosil paste is applied.

Brief Summary Text (14):

This is a method in which the aforementioned chemical agent is applied to the front surface of the teeth, which have been etched, the teeth are rinsed with water after 15 minutes, and they are then polished. In this method, the Aerosil serves as a moisturizing material to prevent the bleaching agent from drying out and further enhances the bleaching effects, resulting in high bleaching effects without the application of light or heat. The 35% H.sub.2 O.sub.2 is highly corrosive and must therefore be handled with care.

Brief Summary Text (16):

In this method, the aforementioned chemical agent is placed on the front of the teeth and allowed to stand to bring about the action for 10 minutes thereof, or the material is irradiated with light for 3 minutes using a visible light ray radiation device. An advantage of this method is that the paste is a light green immediately after being kneaded but turns yellow when irradiated, and when the paste turns brown immediately after being kneaded, this indicates that the bleaching effects of the liquid have diminished. A drawback, however, is the same as that described above with the use of 35% H.sub.2 O.sub.2.

Brief Summary Text (18):

In this method, the aforementioned mixture is allowed to act for 5 minutes on the dental surface, and the teeth are then polished for 15 seconds under light pressure with a polishing disc. These operations are repeated 3 times, the material is then neutralized with 5.25% NaOCl, and the teeth are thoroughly rinsed with water (Oral Surg., 26: 871-878 (1968), J. Am. Dent. Assoc., 87:1329 (1973)). In this method, the paste can scatter into the eyes, making it necessary to adequately protect the patient's eyes.

Brief Summary Text (21):

Many other bleaching methods have also been reported, such as the dental bleaching agent and method comprising a mixture of aqueous hydrogen peroxide and ortho-phosphoric acid (Japanese Laid-Open Patent Application H8-143436/1996), the bleaching agent comprising a mixture of silicic anhydride with aqueous hydrogen peroxide, and the vital bleaching method comprising the application of such a bleaching agent (Japanese Laid-Open Patent Application H5-320033/1993), and the dental bleaching composition comprising a dental bleaching agent (such as hydrogen-urea peroxide, hydrogen peroxide-carbamide, and carbamide peroxide) and a matrix material (such as carboxymethylene), and a method for bleaching teeth using the above (Japanese Laid-Open Patent Application H8-113520/1996).

Brief Summary Text (22):

The following conditions are required of bleaching methods and bleaching agents during dental bleaching, however:

Brief Summary Text (26):

(d) avoidance of detracting from dental physical properties after treatment;

Brief Summary Text (29):

A bleaching method fulfilling the above conditions would be capable of affording cosmetic improvement while preserving dental contour, with considerably improved effects.

Brief Summary Text (30):

In conventional bleaching methods, however, the primary chemical agent is 30 to 35% aqueous hydrogen peroxide, which is highly corrosive, and the oxidative action of which is the basis of bleaching.

Brief Summary Text (31):

All of the various bleaching methods currently used in Japan can be said to comprise, as noted above, a combination of 30 to 35% aqueous hydrogen peroxide, various utensils, and other chemical agents. There is an example of a bleaching method employed in the United States which features the use of 10% urea peroxide instead of 30 to 35% aqueous hydrogen peroxide, but this method is currently embroiled in controversy over problems in terms of efficacy and safety, and has yet to gain approval in Japan.

Brief Summary Text (32):

Bleaching methods covered as insured treatment are currently limited to pulpless teeth, and in some cases pulpectomy is performed on non-carious teeth for the purpose of bleaching. In addition, the use of highly toxic 30 to 35% aqueous hydrogen peroxide in various bleaching methods restricts operations and the like in a variety of ways, while limited bleaching effects for pulped teeth in particular have been indicated.

Brief Summary Text (33):

There is thus a strong need for the development of a safer, faster, simpler novel dental bleaching method that would be effective for both vital teeth and pulpless teeth.

Brief Summary Text (35):

The present invention is intended to provide a method for bleaching discolored teeth using a titanium dioxide photocatalyst.

Brief Summary Text (36):

The present invention relates to a method for bleaching discolored teeth, comprising the steps of applying a solution/paste of titanium dioxide powder and aqueous hydrogen peroxide to the surface of discolored teeth, and bleaching the teeth based on the photocatalytic action that is produced by irradiating this area with light; and also to a bleaching agent which is applied to the surface of discolored teeth to bleach the discolored teeth based on the photocatalytic action that is produced by irradiating this area with light, the aforementioned bleaching agent comprising as active ingredients a combination of aqueous hydrogen peroxide and titanium dioxide producing photocatalytic action when irradiated with light.

Brief Summary Text (37):

Characteristic effects afforded by the present invention include the following: (1) the ability to provide a novel bleaching agent for discolored teeth; (2) the ability to bleach both vital teeth and pulpless teeth; (3) higher safety, better workability, and rapid bleaching effects; (4) alleviation of mental distress on treated patients; (5) the ability to control the level of color adaptation; and (6) the ability to select improvements in color adaptation desired by the treated patient.

Brief Summary Text (39):

As a result of extensive research in light of the foregoing to establish a novel bleaching method that would be safer and simpler, and that would also afford better bleaching effects, the inventors perfected the present invention upon discovering that the intended objectives can be achieved by the combined use of active

ingredients comprising aqueous hydrogen peroxide and titanium dioxide having photocatalytic action.

Brief Summary Text (40):

That is, an object of the present invention is to provide a safer, faster, simpler novel dental bleaching method that would be effective for both vital teeth and pulpless teeth.

Brief Summary Text (41):

A further object of the present invention is to provide a novel bleaching agent used in the aforementioned bleaching method, a method for its manufacture, and a system for bleaching teeth using the aforementioned bleaching agent.

Brief Summary Text (43):

(1) A method for bleaching a discolored tooth, comprising the steps of applying a solution/paste of a titanium dioxide powder and hydrogen peroxide solution onto the surface of the discolored tooth, and irradiating this area with light to bleach the tooth based on the resultant photocatalytic action.

Brief Summary Text (44):

(2) The method for bleaching a discolored tooth according to (1) above, wherein violet or blue visible light is irradiated.

Brief Summary Text (45):

(3) A bleaching agent for applying onto a surface of a discolored tooth to bleach the discolored tooth based on a photocatalytic action that is produced by irradiating this area with light, said bleaching agent comprising as active ingredients a combination of hydrogen peroxide solution and titanium dioxide which produces the photocatalytic action when irradiated with light.

Brief Summary Text (46):

(4) The bleaching agent according to (3) above, consisting of a solution/paste of titanium dioxide with a particle diameter of about 5 to 60 nm and 3% or less hydrogen peroxide solution.

Brief Summary Text (47):

(5) A method for producing a bleaching agent for applying onto a surface of a discolored tooth to bleach the discolored teeth based on the photocatalytic action that is produced by irradiating this area with light, said method for producing a bleaching agent comprising the step of blending hydrogen peroxide solution with a titanium dioxide powder producing photocatalytic action when irradiated with light.

Brief Summary Text (48):

(6) The method for producing a bleaching agent according to (5) above, comprising blending 3% or less hydrogen peroxide solution with a titanium dioxide powder producing photocatalytic action when irradiated with light.

Brief Summary Text (50):

(8) A system for bleaching tooth comprising a combination of a bleaching agent according to (3) or (4) above, means for applying said bleaching agent, irradiating means, and/or other dental treatment materials.

Brief Summary Text (51):

(9) The system for bleaching tooth according to (8) above, wherein a radiation device for generating visible violet light is combined.

Brief Summary Text (53):

To achieve the objects of the present invention, the present invention provides high bleaching effects for discolored teeth, primarily through the oxidative and reductive action of the photocatalyst. In the present invention, the term discoloration is defined as expressing a broad range of meanings including staining.

Brief Summary Text (54):

The chemical agents and utensils used in the present invention may essentially

comprise titanium oxide powder, aqueous hydrogen peroxide, and visible light rays (radiating instrument), affording considerable stability, ease of operation, and bleaching effects. The aforementioned aqueous hydrogen peroxide is more suitably used in lower concentrations, such as a concentration of 6% or less, and preferably a lower concentration of 3% or less.

Brief Summary Text (55):

In a preferred embodiment, the bleaching agent of the present invention is composed of a solution/paste of titanium dioxide powder and aqueous hydrogen peroxide in a low concentration of, for example, 6% or less (and preferably 3% or less). Examples of desirable titanium dioxide include, but are not limited to, titanium dioxide having a particle diameter of about 5 to 60 nm, and particularly anatase type titanium dioxide fine particles or materials based thereon. Any titanium dioxide having similar effects, that is, producing photocatalytic action, can be used, no matter what the shape or properties. Advantages of a smaller particle diameter in this case are that higher activity can be anticipated, good effects can be obtained with lower amounts, lower amounts can be used, high bleaching effects can be obtained more rapidly because thinner coating films can be used, for example, and so forth.

Brief Summary Text (56):

The weight ratio of the aforementioned ingredients can be adjusted as desired depending, for example, on whether the degree of discoloration is lighter or greater, so that products suited to individual cases can be prepared. The bleaching agent of the present invention is ordinarily, but not only, used in the form of a uniform transparent solution or paste by blending, kneading, and dispersing the titanium dioxide powder in aqueous hydrogen peroxide of low concentration. Any type that is prepared in a manner similar to this is encompassed within the range of the present invention. The term "solution/paste" as used in the present invention is defined as any having the aforementioned meaning. In this case, the means and devices for preparing the bleaching agent such as by blending, kneading, dispersion or otherwise manipulating the aforementioned ingredients, as well as the means and the like for applying the bleaching agent, are not particularly limited. Any suitable type can be used. An example of a desirable method for applying the bleaching agent to dental surfaces is to directly coat the dental surface with the bleaching agent.

Brief Summary Text (57):

At this time, fabric, paper, glass cloth, ceramic paper, an organic gel, an inorganic gel, or the like can be impregnated with the bleaching agent of the present invention, that is, a solution or paste of the aqueous hydrogen peroxide and titanium dioxide having photocatalytic action, and the impregnated product can be applied to the dental surface and irradiated with light. Other suitable methods and means can also be used, such as a method in which the aforementioned bleaching agent is supported on a suitable carrier, and is fitted or applied to a tooth or row of teeth.

Brief Summary Text (59):

Discolored teeth can be bleached with the aforementioned bleaching agent by repeatedly applying a solution or paste of a titanium dioxide powder and 3% or less aqueous hydrogen peroxide to a dental surface, for example, and irradiating the coated area. The number of times the material is applied and irradiated may be suitably adjusted according to how light or heavy the staining is. The aforementioned solution or paste should usually be applied by coating or the like using fresh solution or paste about every 15 to 20 minutes. The interval and frequency should be suitably determined according to the state of the teeth.

Brief Summary Text (60):

The bleaching agent of the present invention is effective for bleaching both pulpless teeth and vital teeth, and produces pronounced effects while allowing the teeth to be safely and easily bleached.

Brief Summary Text (61):

The primary action of the bleaching agent in the present invention is bleaching action based on the synergistic action of the titanium dioxide photocatalyst and low

concentration aqueous hydrogen peroxide (for example, 6% or less, and preferably 3% or less, aqueous hydrogen peroxide).

Brief Summary Text (62):

When the titanium dioxide photocatalyst is irradiated with light, electrons and positive holes are produced, reacting with hydrogen peroxide to produce active oxygen. The active oxygen has far greater oxidizing power than ozone, and can oxidize nearly all organic materials into carbon dioxide. Even when n type semiconductor titanium powder with a relatively substantial band gap is used in the form of a solution with 3% aqueous hydrogen peroxide, for example, light radiation readily results in the production of active oxygen having potent oxidizing power, ensuring higher levels of charge separation, electron hole mobility, reactivity with protons or hydroxyl groups, or the like than when used alone, so that synergistic action is produced in addition to the oxidizing action of the 3% aqueous hydrogen peroxide itself.

Brief Summary Text (64):

Dental staining factors are broadly classified into the following extrinsic and intrinsic factors.

Brief Summary Text (75):

II. Bleeding in dental pulp (from external trauma, following pulpectomy, arsenious acid)

Brief Summary Text (77):

IV. Tooth decay, rheumatic fever

Brief Summary Text (86):

The aforementioned staining factors are caused by a variety of pigments, iron salts, tannic acid, chlorhexidine, benzalkonium chloride-chlorhexidine, and cyclones. Such colored substances adhere to tooth enamel and dentine.

Brief Summary Text (87):

A solution of titanium oxide and, for example, 6% or less, and preferably 3% or less, aqueous hydrogen peroxide penetrates between the enamel prisms and dentine, and bleaching is brought about as colored substances are degraded by the oxidative and reductive action of the photocatalyst. The bleaching method of the present invention affords high bleaching effects for discolored teeth caused by both intrinsic and extrinsic factors.

Brief Summary Text (89):

In the present invention, the aforementioned bleaching agent, means for applying the bleaching agent (applicators, etc.), radiation instruments, other chemical agents, other dental treatment materials, instruments, and the like can be suitably combined to produce a system (kit) for bleaching teeth.

Detailed Description Text (3):

0.3 g of anatase type TiO₂ powder with a particle diameter of 7 nm was blended with 1 mL each of a) 0.5% aqueous hydrogen peroxide, b) 1.5% aqueous hydrogen peroxide, and c) 3% aqueous hydrogen peroxide, and the ingredients were kneaded and dispersed to prepare solutions of titanium dioxide and aqueous hydrogen peroxide. 1 g of anatase type TiO₂ powder with a particle diameter of 7 nm was similarly used to prepare a d) paste of 3% aqueous hydrogen peroxide. The products were then housed in containers shielded from light, giving bleaching agents.

Detailed Description Text (4):

(2) Preparation of System for Bleaching Teeth

Detailed Description Text (5):

The aforementioned bleaching agents were housed in containers together with an applicator, radiating instrument (using LED violet light), and preparatory utensils, so as to prepare a system (kit) for bleaching teeth.

Detailed Description Text (6):

(3) Bleaching Discolored Teeth

Detailed Description Text (7):

The aforementioned bleaching agents were used to bleach discolored teeth by the following procedure.

Detailed Description Text (9):

2) The surfaces of the teeth were cleaned by a common method using a rubber cap or the like, and were then dried.

Detailed Description Text (10):

3) The teeth were then temporarily moisture-proofed.

Detailed Description Text (11):

4) The dental surface was coated with a solution of titanium oxide and 3% aqueous hydrogen peroxide, and irradiated with visible light.

Detailed Description Text (15):

Table 1 shows the results obtained with the aforementioned bleaching agents. It is apparent from Table 1 that teeth with mild discoloration (F1) enjoyed considerable bleaching effects after about 1 procedure described above, and that teeth with moderate discoloration (F2 to F3) enjoyed considerable bleaching effects after about 2 to 3 times. Teeth with pronounced discoloration (F4) were bleached after about 4 to 5 times. The aforementioned bleaching effects were long-lasting, with no need for return treatment.

Detailed Description Text (16):

Since the bleaching agent of the present invention provides excellent bleaching effects through the synergism between the bleaching action based on the titanium oxide photocatalyst and the bleaching action of the aqueous hydrogen peroxide, there are no operational restrictions such as those in the case of conventional, highly toxic 30 to 35% aqueous hydrogen peroxide. It may also be understood that the bleaching agent of the present invention can be used for both pulped and pulpless teeth, since it is highly safe.

Detailed Description Text (17):

Compared, in terms of oxidizing energy, to conventional bleaching agents based on aqueous hydrogen peroxide, the bleaching agent in the present invention can be seen to have rapidly resulted in about 2.9 times or more greater bleaching effects.

Detailed Description Text (18):

It is also evident from Table 1 that lower aqueous hydrogen peroxide concentrations tended to require a longer period of time.

Detailed Description Text (19):

The bleaching effects were also enhanced, irrespective of H.sub.2 O.sub.2 concentration, by mixing a solution containing trace amounts of iron salts with the aforementioned bleaching agent applied to the teeth, and then irradiating the coated portion.

Detailed Description Text (30):

(1) Bleaching of Discolored Teeth by Application of Bleaching Agent and Irradiation

Detailed Description Text (31):

Dental surfaces (of extracted teeth) were coated with a bleaching agent (solution) comprising 0.5 g TiO.sub.2 powder mixed with 3% H.sub.2 O.sub.2, and were irradiated with visible light. The coating and irradiation were repeated in the same manner as in Example 1.

Detailed Description Text (32):

As a control, discolored teeth (extracted teeth) were coated with 3% H.sub.2 O.sub.2 by itself, and irradiated with visible light and UV rays continuously for 10 hours.

Detailed Description Text (34):

Following common dental surface cleaning, attempts were made to bleach discolored teeth by applying and irradiating an agent according to a) through c) below.

Detailed Description Text (35):

a) The surface of teeth were coated with the aforementioned TiO_2 -3% H_2O_2 solution using an applicator, and then irradiated with visible light.

Detailed Description Text (36):

b) Dental surfaces were covered with paper ceramic containing TiO_2 powder (by Noritake Company Ltd.). 3% H_2O_2 was then applied thereon using an applicator, and the material was irradiated.

Detailed Description Text (37):

c) Dental surfaces were coated with a paste comprising TiO_2 -3% H_2O_2 (1.2 g/mL) as active ingredients, and were irradiated.

Detailed Description Text (38):

(3) Concentration of Aqueous Hydrogen Peroxide

Detailed Description Text (39):

The concentration of aqueous hydrogen peroxide was varied within the range from 0.1% to 35%, and attempts were made to bleach discolored teeth by applying and irradiating (visible light) the material in the same manner as above.

Detailed Description Text (40):

Extremely discolored teeth were coated on the surface with 30 to 35% H_2O_2 - TiO_2 solutions and irradiated with UV rays.

Detailed Description Text (43):

The type of radiated light (wavelength of 250 to 600 nm) was varied in attempts to bleach discolored teeth by applying and irradiating the material in the same manner as above.

Detailed Description Text (45):

Considerable bleaching effects were noted after about 2 hours in (1) above. Except for discolored teeth resulting from pulp necropathy, the bleaching effects as a whole were excellent. Excellent bleaching effects were also obtained by adjusting the irradiation time according to the degree of discoloration. It was also possible to control adjustments to suitable levels by adjusting the method and time. No bleaching effects were noted in controls.

Detailed Description Text (46):

There were no significant differences in bleaching effects due to bleaching agent configuration or method in (2) above. Technicians can accordingly make their selections by determining the workability depending on the site of the discolored teeth.

Detailed Description Text (50):

As described above, the present invention relates to a bleaching agent for bleaching discolored teeth based on the photocatalytic action that is produced when the agent is applied to the surface of discolored teeth and is irradiated, comprising as active ingredients a combination of aqueous hydrogen peroxide and titanium dioxide producing photocatalytic action when irradiated. The present invention is extremely useful for cosmetic improvements of teeth because of the following effects: (1) the ability to provide a novel bleaching agent for discolored teeth; (2) the ability to bleach both vital teeth and pulpless teeth; (3) higher safety, better workability, and rapid bleaching effects; (4) alleviation of mental distress on treated patients; (5) the ability to control the level of color adaptation; and (6) the ability to select improvements in color adaptation desired by the treated patient.

CLAIMS:

1. A method for bleaching discolored teeth comprising applying a solution or paste or both comprising a titanium dioxide powder and hydrogen peroxide solution onto the surface of the discolored teeth, and irradiating the discolored teeth with light, thereby bleaching the teeth by resultant photocatalytic action.

2. The method for bleaching discolored teeth according to claim 1, wherein said irradiating light is violet or blue visible light.

3. The method for bleaching discolored teeth according to claim 1, which comprises:

a) cleaning surfaces of the discolored teeth;

b) moisture-proofing said cleaned surfaces; and

c) coating said moisture-proofed surfaces with said solution or paste or both of said titanium dioxide powder and said hydrogen peroxide, and irradiating said coated surfaces with visible light.

4. The method for bleaching discolored teeth according to claim 3, wherein step c) is repeated, whereby fresh paste or solution or both is applied and irradiated every 15 to 20 minutes.

5. The method for bleaching discolored teeth according to claim 4, wherein step c) is repeated 2 or 3 times.

6. The method for bleaching discolored teeth according to claim 5, wherein step c) is repeated 4 or 5 times.

7. The method for bleaching discolored teeth according to claim 1, wherein said solution or paste further comprises trace amounts of iron salts.

8. A bleaching composition for bleaching discolored teeth by photocatalytic action produced by irradiation with light, wherein

said bleaching composition comprises as active ingredients a combination of 6% or less hydrogen peroxide solution and crystalline titanium dioxide photocatalyst, wherein

said photocatalyst produces the photocatalytic action and reacts with the hydrogen peroxide to produce active oxygen when irradiated with light.

9. The bleaching composition according to claim 8, which consists essentially of a solution/paste of titanium dioxide with a particle diameter of about 5 to 60 nm and 3% or less hydrogen peroxide solution.

11. A method for producing a bleaching composition for bleaching discolored teeth by photocatalytic action produced by irradiation with light, which method comprises:

blending 6% or less hydrogen peroxide solution with a crystalline titanium dioxide photocatalyst powder, wherein

said photocatalyst producing the photocatalytic action and reacts with hydrogen peroxide to produce active oxygen when irradiated with light.

12. The method for producing a bleaching composition according to claim 11, wherein 3% or less hydrogen peroxide solution is blended with the crystalline titanium dioxide photocatalyst powder.

13. The method for producing a bleaching agent according to claim 11, wherein the crystalline titanium dioxide photocatalyst powder is anatase-type titanium dioxide.

14. A system for bleaching teeth, comprising a combination of a bleaching composition, materials or device for applying said bleaching composition, irradiating device, and optionally other dental treatment materials, wherein the bleaching composition comprises as active ingredients a combination of 6% or less hydrogen peroxide solution and crystalline titanium dioxide photocatalyst,

said photocatalyst producing the photocatalytic action and reacting with the hydrogen peroxide to produce active oxygen when irradiated with light.

15. The system for bleaching teeth according to claim 14, wherein the radiating device generates visible violet light.

16. The system for bleaching teeth according to claim 15, wherein the radiating device generates LED violet light.

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L7: Entry 12 of 106

File: USPT

Apr 3, 2001

US-PAT-NO: 6210163

DOCUMENT-IDENTIFIER: US 6210163 B1

TITLE: Composition and method for cosmetically improving and altering the appearance of teeth

DATE-ISSUED: April 3, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Cohen; Morton	Elkins Park	PA	19027	

APPL-NO: 09/ 370325 [PALM]

DATE FILED: August 9, 1999

PARENT-CASE:

CROSS-REFERENCE TO RELATED APPLICATIONS This application is a continuation-in-part of U.S. application Ser. No. 09/054,898, filed on Apr. 3, 1998.

INT-CL: [07] A61 C 5/00

US-CL-ISSUED: 433/217.1; 106/35

US-CL-CURRENT: 433/217.1; 106/35

FIELD-OF-SEARCH: 433/217.1, 433/215, 433/216, 424/49, 106/35

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

Search Selected

Search ALL

	PAT-NO	ISSUE-DATE	PATENTEE-NAME	US-CL
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ART-UNIT: 372

PRIMARY-EXAMINER: Wilson; John J.

ASSISTANT-EXAMINER: Bumgarner; Melba

ABSTRACT:

A composition and method for altering the appearance of teeth, including whitening or otherwise colorizing teeth, to cover discolorations and stains, or to provide a fashionable color on the tooth which can be selectively removed, the composition comprising a lac based compound solubilized for applying on the surface of a tooth, and a method including selecting the color to be applied, preparing the colorized compound to be applied to a tooth, exposing the tooth to be covered, applying a colorized compound to the enamel surface of the tooth, and allowing the compound to dry on the tooth, and selectively removing the compound from the tooth.

35 Claims, 0 Drawing figures

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L7: Entry 12 of 106

File: USPT

Apr 3, 2001

US-PAT-NO: 6210163

DOCUMENT-IDENTIFIER: US 6210163 B1

TITLE: Composition and method for cosmetically improving and altering the appearance of teeth

DATE-ISSUED: April 3, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Cohen; Morton	Elkins Park	PA	19027	

US-CL-CURRENT: 433/217.1; 106/35

CLAIMS:

What is claimed is:

1. A method for cosmetically altering the appearance of teeth to whiten or colorize the teeth, comprising the steps of:

a) preparing the tooth by exposing the tooth so that the tooth surface to be altered is visible to the gum line;

b) preparing a lac based covering compound to be applied onto the enamel surface of the tooth to be altered, including selecting a compound from a plurality of tooth colored compounds to match the shade of the adjacent or surrounding teeth in the individual's mouth;

c) applying the covering compound prepared in step b. to the tooth surface to be coated; and

d) allowing the covering compound applied to the tooth to dry by exposing the tooth to air.

2. The method of claim 1, wherein the step of preparing a lac based covering compound includes selecting a color from one or more non-tooth colored compounds and mixing said non-tooth-colored compound with said lac based covering compound to provide an overall non-tooth colored appearance, and wherein the covering compound is applied to the tooth surface evenly to cover the tooth surface and is permitted to cure by drying the compound to provide a non-tooth colored coating on the surface of the tooth.

3. The method of claim 2, further including the step of providing palette means, said palette means including a plurality of pigmented compounds, and selecting one or more of said pigmented compounds from said palette means and mixing said selected one or more pigmented compounds with said the lac based covering compound of step b) before applying said lac based covering compound to said tooth.

4. The method of claim 2, further including the step of providing a food grade dye as the colorizing compound and blending said food grade dye with said lac based covering compound to provide a colorized covering compound.

5. The method of claim 1, further including the step of selectively removing the lac based covering compound applied to the tooth.
6. The method of claim 5, wherein the step of selectively removing the lac based covering compound from the tooth includes contacting the compound with a solvent.
7. The method of claim 5, wherein the step of selectively removing the lac based covering compound includes applying to the tooth surface containing the lac based covering compound a forced stream of water.
8. The method of claim 1, further comprising the step of providing colorizing means containing a plurality of pigments to form palette means, and wherein the method further includes the steps of selecting at least one pigment from said palette means, and mixing said selected pigment with said lac based covering compound to be applied to the tooth.
9. The method of claim 1, further including the step of mixing a reflecting material into said lac based covering compound to be applied to said tooth.
10. The method of claim 1, wherein the step of preparing a lac based covering compound includes providing a lac resin and dissolving said lac resin in an ethanol solution.
11. The method of claim 1, wherein the step of preparing the lac based covering compound includes admixing pulverized fluoroaluminosilicate glass particles.
12. The method of claim 1, wherein the step of preparing a tooth further includes the steps of providing a weak acid solution and contacting the tooth surface with the weak acid solution.
13. The method of claim 12, wherein the step of contacting the tooth with a weak acid solution, includes etching the tooth with an acid selected from the group of citric acid and polyacrylic acid.
14. The method of claim 1, wherein the step of preparing a tooth further includes the step of etching microscopic pits which are less than about 5.0 .mu.m in the tooth surface with an etchant.
15. The method of claim 1, wherein the step of preparing a lac based covering compound further includes selecting a food grade dye and blending said dye with said covering compound to provide a colored covering on said tooth.
16. The method of claim 1, wherein the step of preparing a lac based covering compound further comprises providing said lac based covering compound in a fluidic suspension for delivery by spraying, and wherein the step of applying the covering compound to the tooth surface comprises the step of spraying the covering compound on the tooth surface.
17. A method for cosmetically altering the appearance of teeth to whiten or colorize the teeth, comprising the steps of:
 - a) preparing the tooth by exposing the tooth so that the tooth surface to be altered is visible to the gum line;
 - b) providing palette means containing a plurality of tooth-colored shaded pigment compounds, including the step of providing a container means for holding the pigment compounds and providing a mixing area;
 - c) selecting from said palette means one or more of said pigments contained in the container means to match the shade of adjacent or surrounding teeth in an individual's mouth;
 - d) preparing a lac based covering compound to be applied onto the enamel surface of the tooth to be altered by placing said lac based covering compound in said

mixing area of said container means and adding to said lac based covering compound from said container means a pigment compound from said palette means;

e) mixing said covering compound with said pigment compound;

f) applying the lac based covering compound prepared in step e) to the tooth surface to be coated; and

g) drying the covering compound applied to the tooth in step f) by exposing the covered tooth to air.

18. The method of claim 17, wherein the step of preparing a tooth further comprises the steps of:

a) providing a weak acid solution;

b) etching the tooth by contacting the tooth with a weak acidic solution to provide small microscopic pits in the enamel surface of said tooth; and

c) rinsing the weak acid solution off of the tooth with water.

19. A system for cosmetically altering the appearance of teeth wherein a removable coating is applied directly on a dry or wet tooth and cured on the tooth surface, comprising a lac based covering compound having a lac compound and a solvent, one or more colorizing pigment compounds, means for storing the lac based covering compound and associated means for storing one or more colorizing pigment compounds, wherein the lac based covering compound further comprises a compound selected from the group consisting of Bis-GMA sealants and glass ionomeric cements, wherein the system provides the user with the ability to select from the associated means one or more colorizing pigment compounds and mix said selected one or more colorizing pigment compounds with the lac based covering compound and to form a pigmented covering compound, apply the pigmented covering compound to cover the surface of the tooth to be altered, permit the pigmented covering compound to dry by exposure to air, and remove the pigmented covering compound from the tooth at the user's discretion.

20. A composition for cosmetically altering the appearance of teeth to whiten or colorize the teeth by applying said composition to a tooth surface, said composition comprising:

a) a lac resin;

b) a solvent for dissolving said lac resin; and

c) a pigment;

d) wherein the concentration of said lac resin is approximately 1 g/10 ml solvent, and wherein the lac resin is present in the composition in an amount which is greater than the amount of pigment in the composition.

21. The composition of claim 20, wherein said solvent comprises ethanol.

22. The composition of claim 21, wherein said lac resin comprises refined bleached food grade dewaxed lac.

23. The composition of claim 22, wherein said ethanol comprises specially denatured alcohol, formula 45/200 proof.

24. The composition of claim 23, wherein the concentration of said lac resin is approximately 34 to 37% by weight.

25. The composition of claim 20, wherein said pigment comprises titanium dioxide.

26. The composition of claim 20, further including a brightening agent.

27. The composition of claim 26, wherein said brightening agent comprises finely divided fluoroaluminosilicate compound.

28. A covering composition for application to the surface of a tooth for cosmetically altering the appearance of the tooth to whiten or colorize the tooth, said covering composition comprising dewaxed orange flake lac resin solubilized in an ethanol solution at a concentration of 1 g. per 10 ml of ethanol, and a pigment.

29. The covering compound of claim 28, wherein said ethanol solution comprises a 90% ethanol in water.

30. The covering compound of claim 28, wherein said pigment comprises titanium dioxide.

31. The covering compound of claim 28, wherein said pigment comprises a non-tooth colored shade for cosmetically colorizing the tooth when the covering compound is applied thereon.

32. A method for cosmetically altering the appearance of teeth to whiten or colorize the teeth, comprising the steps of:

a) preparing the tooth by exposing the tooth so that the tooth surface to be altered is visible to the gum line;

b) preparing a lac based covering compound to be applied onto the enamel surface of the tooth to be altered, including selecting a compound from a plurality of tooth colored compounds to match the shade of the adjacent or surrounding teeth in the individual's mouth;

c) applying the covering compound prepared in step b) to the tooth surface to be coated;

d) allowing the covering compound applied to the tooth to dry by exposing the tooth to air;

e) wherein the step of preparing a tooth further includes the the steps of providing a weak acid solution and contacting the tooth surface with the weak acid solution; and

f) wherein the weak acid solution is a citric acid of lemon juice.

33. A composition for cosmetically altering the appearance of teeth to whiten or colorize the teeth by applying said composition to a tooth surface, said composition comprising:

a) a lac resin;

b) a solvent for dissolving said lac resin;

c) a pigment;

d) wherein said solvent comprises ethanol; and

e) wherein said lac resin comprises refined bleached food grade dewaxed lac.

34. The composition of claim 33, wherein said ethanol comprises specially denatured alcohol, formula 45/200 proof.

35. The composition of claim 34, wherein the concentration of said lac resin is approximately 34% to 37% by weight.

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L7: Entry 12 of 106

File: USPT

Apr 3, 2001

DOCUMENT-IDENTIFIER: US 6210163 B1

TITLE: Composition and method for cosmetically improving and altering the appearance of teethAbstract Text (1):

A composition and method for altering the appearance of teeth, including whitening or otherwise coloring teeth, to cover discolorations and stains, or to provide a fashionable color on the tooth which can be selectively removed, the composition comprising a lac based compound solubilized for applying on the surface of a tooth, and a method including selecting the color to be applied, preparing the colored compound to be applied to a tooth, exposing the tooth to be covered, applying a colored compound to the enamel surface of the tooth, and allowing the compound to dry on the tooth, and selectively removing the compound from the tooth.

Brief Summary Text (3):

The present invention relates to the field of cosmetically improving and altering the appearance of teeth.

Brief Summary Text (5):

Many procedures are done to improve the appearance of teeth. Teeth are filled to replace dentin and enamel invaded by bacteria, and can be capped to replicate a removed or abraded portion of a tooth.

Brief Summary Text (6):

The teeth of individuals widely vary in their appearance and shape. This is due to the genetic make-up of the individual, but can also be affected by age, and the degree of contact with various foods and medications, both those superficially contacting the teeth and from the internal effects of the medication. The teeth of some individuals exhibit a yellow appearance while those of others may be whiter. While aging is often considered a "natural" cause of tooth discoloration, other factors commonly attributed to tooth discoloration can include chemical exposure to tannins, which are found in red wines, and brewed beverages such as coffees and teas. Additional discoloring chemicals include those not naturally occurring in foods, but rather, manufactured or synthesized compounds, such as, for example, the compounds found in medications, like antibiotics, including tetracycline and other pharmaceuticals. The common practice of using doses of tetracycline to cure facial, acne blemishes has been known to contribute to the discoloration of teeth. Aside from these chemicals, even excess brushing has been can create discoloration by repeated contact with fluoride compounds commonly present in most toothpastes.

Brief Summary Text (7):

Many have attempted to confront the problem of tooth discoloration by proposing various solutions to whiten the teeth. One such method of tooth whitening involves the bonding of veneers onto the buccal or labial surfaces of a tooth. The veneer is usually constructed and applied by a dentist using dental bonding techniques to attach it to the tooth. Various veneers have been described in several U.S. Pat. Nos., see, e.g. 4,992,049 "Method for Applying a Veneer Facing to a Tooth"; 4,822,279 "Article for Cosmetic Restoration of Anterior Teeth" (which uses a glazed porcelain labial veneer); 4,682,950 "Device and Method of Bonding and Veneering Dental Material to a Tooth" (synergizing composite material to a tooth surface which has been etched and coated with a bonding material); 4,473,353 "Method for Cosmetic Restoration of Anterior Teeth" (wherein a glazed porcelain veneer is bonded to a patient's tooth); and 4,433,959 "Composite Laminate Dental Veneer Containing Color

Systems" (a veneer which is molded and then attached to the labial enamel surfaces of teeth).

Brief Summary Text (8):

Other attempts to whiten teeth are also known to include bleaching the teeth. Often the bleach is applied in the form of hydrogen peroxide, which can be obtained in drug stores by consumers. Because of the delicacy of applying hydrogen peroxide in one's mouth, some dentists carry out the procedure in their offices, using a stronger peroxide than can be purchased by the consumer. There are even pastes, sold over the counter, to the consumers which claim to whiten teeth. Often ordinary toothpastes make this claim, but increasingly appearing in the marketplace for purchase by consumers are pastes with the chemical compound sodium bicarbonate (baking soda), which may also contain peroxide. While chemical bleaching of teeth has been done to provide whiter-looking teeth, with its use there exists danger to the enamel of the teeth, especially if excessive exposure to chemical bleaches occurs. Further, chemical bleaching is understood in many cases to require multiple applications, and, hence, repeated use of the chemical. Even when applied by a dentist, precautions may be taken to prevent peroxide solution from contacting the patient's gums, which if otherwise allowed to come into contact therewith can be painful and cause damage to the gums. In cases of certain stains, bleaching may not be effective, and the stain may remain.

Brief Summary Text (9):

There are even procedures involving abrasion of the tooth enamel to present a smooth surface which is lighter in appearance than the stained surface removed. This has limitations as to the number of times it can be done.

Brief Summary Text (10):

Furthermore, abrading or bleaching teeth can have deleterious side effects, including, increased sensitivity of the treated tooth to temperature, i.e. especially when hot and cold foods and drinks are consumed. This effect may subside within time, but often the need to repeat bleaching procedures regularly, gives rise to a period of time within which the treated tooth can be hypersensitive.

Brief Summary Text (11):

There are some prior art whitening methods which require etching steps that are carried out with phosphoric acids. The use of phosphoric acid is generally done by a dentist under controlled conditions, for example, in the dentist's office. Caustic acid etchants have been recognized to be corrosive to the soft tissues of the mouth. For example, ortho-phosphoric acid, in some venues, must be transported pursuant to specified requirements and restrictions. In addition, the long term physiological effects of acid etching, which are generally unknown, have led practitioners to question certain acid etching uses in the field of dentistry. See e.g. M. G. Buonocore, "The Challenge of Bonding to Dentin", The Acid Etch Technique, L. M. Silverstone and I. L. Dogon, Eds., Proceedings of the International Symposium at St. Moritz, Switzerland, Dec. 16-18, 1974, North Central Publishing Co. (St. Paul, 1975). See also, U.S. Pat. No. 5,304,585, which raises these concerns, the complete disclosure of which is herein incorporated by reference; and see Akira Komori, and Haruo Ishikawa, "Evaluation of a Resin-Reinforced Glass Ionomer Cement for Use as an Orthodontic Bonding Agent," The Angle Orthodontist, Vol. 67 No. 3, 1997, the complete disclosure of which is herein incorporated by reference. Further attempts to whiten teeth are disclosed in U.S. Pat. Nos. 4,032,627 "Tooth Whitening Cosmetic Composition"; 4,097,994 "Dental Restorative Composition Containing Oligomeric Bis-GMA Resin and Michler's Ketone"; 4,141,144 "Dental Material and Method For Controlling Tooth Lustre"; and 4,512,743 "Method for Masking Discoloration on Teeth." U.S. Pat. Nos. 4,512,743 and 4,141,144, each use phosphoric acid application to the tooth in their treatments. U.S. Pat. No. 4,097,994 discloses a photocurable compound, which is used with a specific ultraviolet sensitizer and a peroxide catalyst to cure the compound. Furthermore, phosphoric acid etching generally disposes grooves in the tooth enamel in the nature of about 50 to 60 .mu.m. This order of etching is visible and is noticed in the form of a dull tooth surface.

Brief Summary Text (12):

U.S. Pat. No. 4,032,627, referenced above, discloses the use of an alcohol-soluble composition to be applied to the surface of a tooth to whiten the tooth's

appearance. This composition is suggested to be applied by the user. However, although this disclosure attempts to provide an extended wearing time for its compound, the composition is readily worn off by the abrasive action of food eaten after the compound is applied to the teeth, with certain, more abrasive, harder, foods causing faster wear of the composition from the tooth than other, softer, foods.

Brief Summary Text (13):

A need exists for a temporary tooth whitening and colorizing system which can be safely applied by the wearer, or without the need for specially trained dental personnel. The tooth whitening to be achieved should also have stability and be able to resist removal or solvation by foods and beverages, but should be able to be removed whenever the user desires. It is further desirable to provide a composition and a method for covering a tooth, to appear white or colorized, which can be selectively applied and removed by a wearer without harming the tooth.

Brief Summary Text (15):

A novel composition and method for altering the appearance of teeth is provided by the present invention, where an individual, in his or her own home, or in a beauty parlor, or other non-medical office, can apply a colorized compound to his or her teeth to cosmetically alter and/or improve the appearance of the teeth. The present method can be used to whiten anterior teeth, and can even be done in the presence of existing saliva which may be present on the tooth. The method provides a removable coating which can comprise a colorized coating which can be removed and replaced with other colorized coatings, or no further coatings, at the user's discretion. Expensive or dangerous drying apparatus are not required. Further, the present invention can be applied with or without an etching step, thereby avoiding the hazards inherent to use of caustic acid etchants, such as phosphoric and phosphonic acids. Moreover, when an individual's teeth, in accordance with the present method, require pre-treatment prior to application of the covering compound, the application of a naturally occurring substance can be used to facilitate adhesion. The substance can be lemon juice or lime juice, which unlike caustic acids, contains citric acid, which can be purchased by the consumer user of the covering compound which is to be applied by the present method. In a proposed alternate embodiment of the invention, polyacrylic acid is applied to the tooth surface prior to contact with the covering compound.

Brief Summary Text (16):

The present invention also provides a novel method for temporarily changing the color of a tooth. A palette system is provided wherein a user or wearer can select a color from one, or a combination of one or more, colorizing compounds. The selected or created color can then be incorporated with the other steps of the present method to provide a unique appearance to a tooth. The present method permits the user to mix his or her own colors or to select a color from one or more prepared provided colors.

Brief Summary Text (17):

The composition is a removable compound which can be applied to a tooth surface. Preferably, the composition includes a lac material, which is provided with a colorizing regulating compound or substance, in a solvent such as ethanol. Lac, the natural resinous substance excreted by an insect, Laccifer Lacca, has been used in dentistry mainly for the construction of special trays and bases for wax rims when recording jaw relationships. (See A. Azucca, R. Huggett, and A. Harrison, "The Production of Shellac and its General and Dental Uses: A review." Journal of Oral Rehabilitation, 1993, vol. 20, pp. 393-400, the complete disclosure of which is herein incorporated by reference; and I. Klineberg and R. Earnshaw, "Physical Properties of Shellac Baseplate Materials." Australian Dental Journal, October, 1967, vol. 12 no.5, pp. 468-475.) Another use of shellac in dentistry includes treatment of a cavity with a hydrophilic shellac film placement of a polystyrene liner. (See M. Blixt and P. Coli, "The Influence of Lining Techniques on the Marginal Seals of Class II Composite Resin Restorations" Quintessence International, vol. 24, no.3, 1993). Shellac has also been prepared and used in dentistry for the use of a bead adhesive for securing a composite resin veneer cast restoration. (See C. Lee, H. Pierpont, and E. Strickler, "The Effect of Bead Attachment Systems on Casting Patterns and Resultant Tensile Bond Strength of Composite Resin Veneer Cast

Restorations", The Journal of Prosthetic Dentistry, November, 1991, vol. 66, no.5, pp. 623-630.)

Brief Summary Text (18):

It is an object of the present invention to provide a novel composition and method for improving the appearance of teeth.

Brief Summary Text (19):

It is a further object of the present invention to provide a composition and a method that can be useful for whitening the teeth to improve or alter their appearance.

Brief Summary Text (20):

It is a further object of the present invention to improve or alter the appearance of teeth by covering stains or discolorations on a tooth by matching a colored compound to an individual's teeth and applying the colored compound on the surface of said stained or discolored teeth.

Brief Summary Text (21):

A further object of the present method is to improve the appearance of a tooth by applying a colored or shaded composition to the tooth.

Brief Summary Text (22):

A further object of the present invention is to coordinate colors of a tooth with other health and beauty aids by applying a method for colorizing a compound and applying it to a tooth.

Brief Summary Text (23):

Another object of the present invention is to provide a method for applying a compound to a tooth which can remain on the tooth during eating and other activity, and can be removed at the wearer's discretion, to leave the tooth as it appeared before the application of the compound.

Brief Summary Text (24):

A further object of the present invention is to provide a novel method of improving the appearance of a tooth which includes applying a compound to the tooth and selectively removing the compound from the tooth.

Brief Summary Text (25):

A further object of the present invention is to provide a method for improving the appearance of a tooth which can be repeated by the wearer, to change the appearance of a tooth, on a regular basis if the wearer desires, without having damaging effects on the tooth enamel.

Brief Summary Text (26):

Another object of the present invention is to provide a method which includes a color matching system wherein the user mixes components to colorize the composition to be applied to the tooth to the desired hue or color, which can be a tooth color or a non-tooth color.

Brief Summary Text (27):

It is another object of the present invention to accomplish the above objects by repeating application and removal of the compound on a tooth for different colors as the user determines and selects.

Brief Summary Text (28):

Another object of the present invention is to provide a tooth covering composition which can be selectively colorized, applied and removed by a user.

Brief Summary Text (29):

Another object of the present invention is to provide a compound which can be selectively removed from the tooth by a forced stream of water.

Brief Summary Text (31):

The present invention provides a method for altering the appearance of teeth. The

altered appearance can be the removal of stains or discoloration caused by disease, chemical exposure or aging, or can be the application of a color (i.e. red, blue green, etc) to the tooth. Generally the enamel of teeth is bound to tannins and other chemicals which remain on the enamel, in the form of a stain, which cannot be removed by brushing.

Brief Summary Text (32):

The present invention provides a composition which can be selectively applied to a tooth surface to provide a cosmetic alteration of the tooth, specifically, whitening or colorizing the tooth. The compound, which is a lac based compound, preferably can be provided, or can be mixed by the user, to colorize the tooth a tooth colored shade, or one or more of a variety of colors (i.e. red, blue, green etc.). The present method provides for the application of a lac based compound to the surface of a tooth to cover discolorings which are present on the tooth, or to provide a colorization of the tooth.

Brief Summary Text (33):

A pigment compound is used to regulate the color of the tooth which is to be covered with the covering compound. For example, one pigment which can be used is titanium dioxide, which can yield a white or tooth colored shade. Other suitable pigments can be used, either separately, or combined to form desired shades.

Brief Summary Text (34):

The covering compound to be applied by the present method comprises an opaque material which masks the tooth surface. Preferably, the covering compound comprises a lac based compound.

Brief Summary Text (35):

The compound is prepared by dissolving a lac material into a solvent. The solvent used for the example below is ethanol (99% ethyl alcohol). The compound is preferably a liquid form which is fluid and can be painted on the surface of a tooth.

Brief Summary Text (41):

The covering compound is applied to the teeth in the present invention by painting it onto the tooth surface, preferably with a brush. This enables the composition to be evenly distributed to cover the entire tooth, and furthermore, without an excessive buildup or absence of material in one spot of the tooth. Alternately an aerosol suspension can be used to apply the covering compound to the tooth. For example, a small tube can be used through which the aerosol can be propelled for application onto the tooth. This facilitates even distribution of the covering compound on the tooth surface and avoidance of coverage on gums, skin and other non-tooth surfaces.

Brief Summary Text (42):

The present method also provides palette means for controlling the colorizing of the tooth. The palette means preferably comprises a plurality of pigments which are present in individual quantities for adding to the covering composition to be applied to the tooth. The palette means preferably is provided with a plurality of pigment means which contain tooth-colored pigments which can be matched to the individual user's tooth color. In addition, the palette means can contain colorful shades, such as, for example, blue, pink, pastel colors, or any other color which is supplied in the form of a pigment compound which can be mixed with the base covering compound and then be applied to the tooth by painting on the tooth.

Brief Summary Text (43):

The method can be carried out, for example, by providing a tray having a series of compartments containing tooth-colored pigments arranged in one row and a series of colorizing pigments in other rows. A larger compartment can be provided for the base compound or components. The compartments can have resealable covers which can be opened and closed by the user to remove a pigment or compound as needed. A mixing area can also be provided and can also have a cover.

Brief Summary Text (44):

Alternately, it is possible to apply the lac-based covering compound on the tooth,

and then mix the color or pigment compound with the lac compound directly on the tooth. This may be done, for example, to facilitate approximating the shade of adjacent teeth.

Brief Summary Text (45):

In some circumstances, it may be desirable to etch a tooth surface to provide microscopic pits for facilitating adhesion of a compound to be applied thereto. Etching provides an increased surface area of the tooth. However, the present method also requires removability of the temporary tooth coating. In a preferred embodiment of the invention, the teeth of an individual can be pretreated with citric acid, of the same weakness of that concentration contained in lemon juice. This facilitates the adherence of the covering compound on the tooth's surface. The citric acid prepares the surface of the tooth to receive the compound which is to be painted on the tooth.

Brief Summary Text (46):

The method contemplates application of the tooth enhancing composition by an individual user or wearer having no special training or knowledge in dentistry. In this case, lemon juice can be used, since it will be easily obtained by the user. Alternately, citric acid solutions can be prepared or provided having the same general concentration as citric acid in lemon juice. By the use of the etching step, very small grooves or pits are formed on the tooth surface, which are approximately 5 to 10 .mu.m or less. This enables the wearer of the compound to restore a treated tooth to its original appearance when desired by removing the covering compound. Another acid proposed for use with the compound of the present invention, for tooth preparation is polyacrylic acid.

Brief Summary Text (47):

Alternately, it is proposed that the covering compound, for example, can be prepared by taking a lac based composition, such as of the type described in Examples 1 or 2, comprising a 1 g. lac/10 ml ethanol solution, or in Examples 3 or 4, and providing an amount of a pigment compound which becomes the base pigment. To this pigmented base compound, the user can customize the color with the colorizing means, by selecting and combining pigment items and admixing the selected pigment items with the base compound. In addition, other elements, such as reflective means can be admixed as well to provide additional unique alteration of the tooth. For example, polymer particles, such as glitter, can be added to provide a sparkle effect to the tooth.

Brief Summary Text (48):

A colorizing pigment can be supplied in the form of a modified food starch or other color additive. The pigment can be supplied with the lac compound, or can be provided separately to be admixed by the user with the lac compound. The lac compound can be supplied to the user with a pigment to exhibit a tooth-colored shade, and can even be further mixed for colorization by the user.

Brief Summary Text (50):

The form of the additive for use in the present invention preferably includes dye form additives, but may also include lake forms which are compatible with the lac based covering composition. Water soluble dyes, provided in the form of powders, granules, liquids or other special-purpose forms can be used in accordance with the present method. Lakes, the water insoluble form of the dye, are generally used for coloring products which do not contain adequate moisture to dissolve the dyes. For example, if a suspension of color is to be used, a lake form additive can be employed. The color additive provided in the form of a lake may, for example, be used with other tooth appearance enhancing means such as glitter particles.

Brief Summary Text (51):

The present method includes the step of painting the compound on a tooth which is in its in vivo environment and an intermediary is not required. The naturally occurring saliva may be present on the tooth, and the compound can be applied with the saliva being present. Alternately, the method can include applying the compound to a tooth by spraying. For example, a compressed gas propellant, such as an aerosol, can be utilized to provide delivery of the covering compound to the tooth.

Brief Summary Text (52):

The selected compound is painted on the tooth and permitted to harden. The hardening of the compound occurs within about two to three minutes, wherein the alcohol dries or evaporates and the solubilized lac composition polymerizes. A layer is then formed by the compound covering the tooth surface on which it was applied, and becomes fixed on the tooth. The tooth thereby exhibits a new appearance, attributable to the compound.

Brief Summary Text (53):

The compound exhibits permanence and withstands normal buccal functions such as, for example, brushing, eating, chewing, contacting foods and beverages, and other functions carried out with one's teeth. The method applies a covering on the tooth which maintains a uniform appearance and is further resistant to staining. In addition, the covering protects the tooth against further staining and contact with bacteria and chemicals. The method applies a thin coating to the tooth which does not interrupt the user's normal mouth functions, and does not feel uncomfortable to a user.

Brief Summary Text (54):

The method further includes selectively removing the coating. The coating is removably provided on the tooth and can be removed from the tooth at the user's discretion. For example, if a user desires to change the color of the coated tooth, and, for example, apply a different color, then the first coating is removed to expose the original tooth surfaces again. A second or next coating can then be applied to the tooth to change the color. Removing preferably includes the step of using a pressurized stream of water, which can be done with a commercially available appliance, such as, for example, a WATER PIK.RTM..

Brief Summary Text (55):

Alternately, it is proposed that a solvent may be used to remove the tooth coating. Preferably, the solvent comprises a composition which the user can readily obtain, or which can be supplied to the individual, non-medical personnel user. Furthermore, it is conceivable that compatible solvents can be used for removing the covering compound from a tooth by softening or dissolving the compound or its bonds. The solvent, for example, can be a material which may break up bonds between the lac polymerization and/or invade a bond formed between the compound and the tooth surface. The removal step wherein a solvent is used, can be accomplished by swabbing the solvent onto the tooth coating or around the edges thereof with a cotton swab. The coating is then loosened and can be more easily removed, or can even be dissolved.

Brief Summary Text (56):

Removal of the covering coating from the tooth then displays the original surface of the underlying tooth which had been covered by the coating.

Brief Summary Text (57):

The following is an example of the use of the lac based covering compound and the method of the present invention, as carried out on human teeth in vivo. The dewaxed orange flake lac resin was used as indicated in Examples 1 and 2, below, to formulate a covering compound for cosmetically altering the appearance of a tooth, although it is conceivable that other lac resins, such as white lac, can also be used consistent with the scope of the present invention. While ethanol is described as a preferred solvent, it will be understood that an ethanol solution of less than 99% pure ethanol may be utilized, however, the drying times may be affected by the addition of alternate solvents.

Detailed Description Text (2):

An anterior tooth was prepared by retracting the individual's lip to expose the entire tooth, up to and including the upper gum line. The tooth was a front upper tooth. A quantity of 1 g. of lac resin (obtained commercially from A.F. Suter & Co. Ltd. as "Dewaxed Orange Flake Shellac" as discussed above), a dry light brown flake material was dissolved in 10 ml of grain alcohol (99% ethanol). A coloring agent was then added to tint the lac mixture to the desired shade. In this example, titanium dioxide powder (obtained commercially from Gamblin Dry Pigments, P.O. Box 625, Portland, Oreg. 97207) was admixed with the lac solution. The amount of titanium

dioxide powder brought the color of the amber solution to a tooth colored shade, and the addition was an amount sufficient to arrive at the desired shade.

Detailed Description Text (3):

The covering compound was then applied onto the surface of the tooth. The covering compound dried within two minutes of its application by exposure to the air, the alcohol evaporating from the solution leaving a lac coating on the tooth. The result was an evenly-coated tooth, which presented an evenly-colored appearance. Any discoloration or uneven shading which was previously present on the tooth was no longer visible.

Detailed Description Text (4):

The tooth was used normally, for eating, drinking, and was brushed regularly for a four-day period, after which the coating on the tooth was easily removed by the application of a high pressure stream of water (supplied by the use of a WATER PIK.RTM., an instrument which applies a pressurized stream of water onto the tooth, and which is commercially available to consumers). The coating was abraded and removed from the tooth with a WATER PIK.RTM..

Detailed Description Text (6):

The above conditions were repeated, as reported for Example 1, above, for an upper front tooth. However, the tooth was first prepared by exposing the tooth to citric acid (applied in the form of lemon juice). The lemon juice was permitted to remain on the tooth for one minute, after which time, it was washed off of the tooth with a water rinse. The lac based covering compound was then prepared and applied to the washed tooth, which was still wet, in accordance with the same procedure as in Example 1, above. This coating performed, and could be removed, in the same manner as the coating in Example 1, above.

Detailed Description Text (8):

An upper front tooth was prepared by retracting the individual's lip to expose the entire tooth, up to and including the upper gum line. The tooth was a front upper tooth. A quantity of about 0.10 mls of a lac resin solution (containing bleached food grade (USP) dewaxed lac resin solubilized in specially denatured alcohol formula 45/200 proof (SDA 45/200), obtained commercially from Mantrose-Hauser Company, 1175 Post Road East, Westport, Conn. 06880, USA) was taken on a sable brush. The solution was a clear color.

Detailed Description Text (9):

A coloring agent was then added to tint the lac solution to a desired shade. In this example, titanium dioxide powder (obtained commercially from Gamblin Dry Pigments, P.O. Box 625, Portland, Oreg. 92707) was admixed with the sample of lac solution taken on the brush. The sable brush deposited the lac solution sample onto a clean dish. An amount of pigment, equal to the size of a pinhead was added to the dish containing the lac solution sample were admixed using the brush, until a uniformly pigmented covering compound result (i.e. wherein the pigment was uniformly distributed throughout the compound). The amount of titanium dioxide powder brought the color of the clear solution to a tooth colored shade, and the addition was an amount sufficient to arrive at the desired shade. The covering compound was brushed onto the tooth surface. The covering compound dried within minutes of its application. The tooth was used normally, for eating, drinking, and was brushed regularly for a four-day period, after which the coating on the tooth was easily removed by the application of a high pressure stream of water (supplied by the use of an instrument which applies a pressurized stream of water onto the tooth, and which is commercially available to consumers, such as, for example; a WATER PIK.RTM.). The coating was abraded and removed from the tooth with the WATER PIK.RTM..

Detailed Description Text (11):

The above conditions were repeated, as reported for Example 3, above, for a front upper tooth. However, the tooth was first prepared by exposing the tooth to citric acid (applied in the form of lemon juice). The lemon juice was permitted to remain on the tooth for one minute, after which time, it was washed off of the tooth with a water rinse. The lac based covering compound was then prepared and applied to the washed tooth, which was still wet, in accordance with the same procedure as in

Example 3, above. This coating performed, and could be removed, in the same manner as the coating in Example 3, above.

Detailed Description Text (13):

The above conditions were repeated, as reported for Example 3, above, for an upper front tooth. The covering compound was prepared by taking a quantity of about 0.10 mls of a lac resin solution containing bleached food grade (USP) dewaxed lac resin solubilized in specially denatured alcohol formula 45/200 proof (SDA) 45/200, (obtained commercially from Mantrose-Hauser Company, 1175 Post Road East, Westport, Conn. 06880, USA) which was taken on a brush and mixed with a quantity of pigment in a dish. The preparation of the covering compound was in accordance with the procedure identified above in Example 3, however, instead of titanium dioxide powder, a red pigment (red # 22, sold commercially and obtained from Warner Jenkinson Co. Inc., 107 Wade Avenue, South Plainfield, N.J. 07080 as K-7008) was used. The compound was mixed and a red color resulted. The red coloring compound was applied to a tooth in the manner described in connection with Example 3. The tooth, having the red color from the compound applied to it, was used normally, for eating, drinking and was brushed regularly for a four-day period, after which the coating on the tooth was easily removed by the application of a high pressure stream of water (supplied by the use of an instrument which applies a pressurized stream of water onto the tooth and which is commercially available to consumers, such as, for example, a WATER PIK.RTM.). However, the tooth was first prepared by exposing the tooth to citric acid (applied in the form of lemon juice). The lemon juice was permitted to remain on the tooth for one minute, after which time, it was washed off of the tooth with a water rinse. The lac based covering compound was then prepared and applied to the washed tooth, which was still wet, in accordance with the same procedure as in Example 3, above. This coating performed, and could be removed, in the same manner as the coating in Example 3, above.

Detailed Description Text (14):

This example was also repeated using, in place of the red pigment, yellow # 6 powder (FD&C 08006), and again using D & C yellow #10 (K-7059) (Warner-Jenkinson). Similar results were obtained, however instead of a red color on the tooth, a yellow or orange color was obtained.

Detailed Description Text (17):

The covering compound is initially prepared, in accordance with Example 1, or by starting with the lac solution of Example 3. The covering compound is then further prepared by admixing with it a colorizing pigment. The colorizing pigment can be selected from color additives, including titanium dioxide and other pigments, vegetable dyes and the like. In this manner a variety of tooth-colored shades are possible, and can be matched to existing or surrounding teeth shades, by approximation with the addition of a pigment.

Detailed Description Text (18):

In this example, it is proposed that the covering compound is provided in a tooth colored shade to the user. The provided covering compound is then admixed with a selected pigment to provide a colorized compound. The colorized compound is applied to a tooth surface in the manner recited above in Examples 1 or 3, by painting it on the tooth surface. The compound is then permitted to dry by exposure to air, and selectively removed at the wearer's discretion.

Detailed Description Text (21):

The lac based covering compound can be provided in a tooth colored shade which can be further customized by the user with a selection from the pigment compounds. The tooth colored shade can preferably be provided by the addition of titanium dioxide or other suitable pigment, or by varying the types of lac used, or both.

Detailed Description Text (22):

The colorized covering compound is applied to a tooth surface in the manner recited above in Examples 1 or 3. The covering compound is then permitted to dry by exposure to air, and selectively removed at the wearer's discretion.

Detailed Description Text (24):

The compounds and methods described in Examples 1, 3, 5, 6 and 7, above, but further

including an etching step. A weak acid is provided to etch very small microscopic pits onto the tooth surface. A citric acid solution is used. The citric acid solution preferably has the same concentration as lemon juice and, further, can be used in the form of lemon juice. The lemon juice is applied onto the tooth surface which is to receive the covering compound and allowed to remain on the tooth from about a few seconds to a few minutes. The lemon juice is then washed from the tooth with a water rinse. The tooth, still wet, is now ready to receive the covering compound. The covering compound is then applied by brushing onto the tooth surface to provide an evenly dispersed coating on the tooth. The compound is then permitted to dry by exposure to air, and selectively removed at the wearer's discretion.

Detailed Description Text (26):

The method is carried out as in Example 8, wherein the tooth preparing step includes etching the tooth with a polyacrylic acid solution (in place of the citric acid solution) by contacting the tooth with the polyacrylic acid solution and allowing the acid solution to remain on the tooth for a couple of minutes. The polyacrylic acid solution is then rinsed off of the tooth by applying a water rinse. Thereafter, the covering compound is applied.

Detailed Description Text (28):

The method is carried out as in any of Examples 1-9 above, wherein the covering compound is provided having a powdered component and a liquid component. The powdered component can contain the lac base and a pigment, which can be a tooth colored pigment or a non-tooth colored pigment. The powdered component can be provided in a plurality of pigmented shades for selection by the user. The powdered component is selected and mixed with a liquid component, which, for example, can contain ethanol, to form the lac based covering compound for application to an etched or non-etched tooth.

Detailed Description Text (31):

In each of the above examples, it is noted that no effort is made to dry the tooth, prior to applying the covering compound thereon.

Other Reference Publication (1):

Klineberg I. And Earnshaw R., "Physical Properties of Shellac Baseplate Materials." Australian Dental Journal. Oct., 1967, vol. 12, No. 5, pp. 468-475.

Other Reference Publication (2):

Azouka A., Hugget R., and Harrison A. "The Production of Shellac and its General and Dental Uses: A Review." Journal of Oral Rehabilitation. 1993, vol. 20, pp. 393-400.

Other Reference Publication (8):

Silverman E., Cohen M., Demke, and Silverman M., "A New Light-Cured Glass Ionomer Cement That Bond Brackets to Teeth Without Etching in the Presence of Saliva", (American Association of Orthodon.), 1995, vol. 108, pp. 231-236.

CLAIMS:

1. A method for cosmetically altering the appearance of teeth to whiten or colorize the teeth, comprising the steps of:

- a) preparing the tooth by exposing the tooth so that the tooth surface to be altered is visible to the gum line;
- b) preparing a lac based covering compound to be applied onto the enamel surface of the tooth to be altered, including selecting a compound from a plurality of tooth colored compounds to match the shade of the adjacent or surrounding teeth in the individual's mouth;
- c) applying the covering compound prepared in step b. to the tooth surface to be coated; and
- d) allowing the covering compound applied to the tooth to dry by exposing the tooth to air.

2. The method of claim 1, wherein the step of preparing a lac based covering compound includes selecting a color from one or more non-tooth colored compounds and mixing said non-tooth-colored compound with said lac based covering compound to provide an overall non-tooth colored appearance, and wherein the covering compound is applied to the tooth surface evenly to cover the tooth surface and is permitted to cure by drying the compound to provide a non-tooth colored coating on the surface of the tooth.
3. The method of claim 2, further including the step of providing palette means, said palette means including a plurality of pigmented compounds, and selecting one or more of said pigmented compounds from said palette means and mixing said selected one or more pigmented compounds with said the lac based covering compound of step b) before applying said lac based covering compound to said tooth.
5. The method of claim 1, further including the step of selectively removing the lac based covering compound applied to the tooth.
6. The method of claim 5, wherein the step of selectively removing the lac based covering compound from the tooth includes contacting the compound with a solvent.
7. The method of claim 5, wherein the step of selectively removing the lac based covering compound includes applying to the tooth surface containing the lac based covering compound a forced stream of water.
8. The method of claim 1, further comprising the step of providing colorizing means containing a plurality of pigments to form palette means, and wherein the method further includes the steps of selecting at least one pigment from said palette means, and mixing said selected pigment with said lac based covering compound to be applied to the tooth.
9. The method of claim 1, further including the step of mixing a reflecting material into said lac based covering compound to be applied to said tooth.
12. The method of claim 1, wherein the step of preparing a tooth further includes the steps of providing a weak acid solution and contacting the tooth surface with the weak acid solution.
13. The method of claim 12, wherein the step of contacting the tooth with a weak acid solution, includes etching the tooth with an acid selected from the group of citric acid and polyacrylic acid.
14. The method of claim 1, wherein the step of preparing a tooth further includes the step of etching microscopic pits which are less than about 5.0 .mu.m in the tooth surface with an etchant.
15. The method of claim 1, wherein the step of preparing a lac based covering compound further includes selecting a food grade dye and blending said dye with said covering compound to provide a colored covering on said tooth.
16. The method of claim 1, wherein the step of preparing a lac based covering compound further comprises providing said lac based covering compound in a fluidic suspension for delivery by spraying, and wherein the step of applying the covering compound to the tooth surface comprises the step of spraying the covering compound on the tooth surface.
17. A method for cosmetically altering the appearance of teeth to whiten or colorize the teeth, comprising the steps of:
- a) preparing the tooth by exposing the tooth so that the tooth surface to be altered is visible to the gum line;
 - b) providing palette means containing a plurality of tooth-colored shaded pigment compounds, including the step of providing a container means for holding the pigment compounds and providing a mixing area;

- c) selecting from said palette means one or more of said pigments contained in the container means to match the shade of adjacent or surrounding teeth in an individual's mouth;
- d) preparing a lac based covering compound to be applied onto the enamel surface of the tooth to be altered by placing said lac based covering compound in said mixing area of said container means and adding to said lac based covering compound from said container means a pigment compound from said palette means;
- e) mixing said covering compound with said pigment compound;
- f) applying the lac based covering compound prepared in step e) to the tooth surface to be coated; and
- g) drying the covering compound applied to the tooth in step f) by exposing the covered tooth to air.

18. The method of claim 17, wherein the step of preparing a tooth further comprises the steps of:

- a) providing a weak acid solution;
- b) etching the tooth by contacting the tooth with a weak acidic solution to provide small microscopic pits in the enamel surface of said tooth; and
- c) rinsing the weak acid solution off of the tooth with water.

19. A system for cosmetically altering the appearance of teeth wherein a removable coating is applied directly on a dry or wet tooth and cured on the tooth surface, comprising a lac based covering compound having a lac compound and a solvent, one or more colorizing pigment compounds, means for storing the lac based covering compound and associated means for storing one or more colorizing pigment compounds, wherein the lac based covering compound further comprises a compound selected from the group consisting of Bis-GMA sealants and glass ionomeric cements, wherein the system provides the user with the ability to select from the associated means one or more colorizing pigment compounds and mix said selected one or more colorizing pigment compounds with the lac based covering compound and to form a pigmented covering compound, apply the pigmented covering compound to cover the surface of the tooth to be altered, permit the pigmented covering compound to dry by exposure to air, and remove the pigmented covering compound from the tooth at the user's discretion.

20. A composition for cosmetically altering the appearance of teeth to whiten or colorize the teeth by applying said composition to a tooth surface, said composition comprising:

- a) a lac resin;
- b) a solvent for dissolving said lac resin; and
- c) a pigment;
- d) wherein the concentration of said lac resin is approximately 1 g/10 ml solvent, and wherein the lac resin is present in the composition in an amount which is greater than the amount of pigment in the composition.

25. The composition of claim 20, wherein said pigment comprises titanium dioxide.

28. A covering composition for application to the surface of a tooth for cosmetically altering the appearance of the tooth to whiten or colorize the tooth, said covering composition comprising dewaxed orange flake lac resin solubilized in an ethanol solution at a concentration of 1 g. per 10 ml of ethanol, and a pigment.

30. The covering compound of claim 28, wherein said pigment comprises titanium dioxide.

31. The covering compound of claim 28, wherein said pigment comprises a non-tooth colored shade for cosmetically colorizing the tooth when the covering compound is applied thereon.

32. A method for cosmetically altering the appearance of teeth to whiten or colorize the teeth, comprising the steps of:

- a) preparing the tooth by exposing the tooth so that the tooth surface to be altered is visible to the gum line;
- b) preparing a lac based covering compound to be applied onto the enamel surface of the tooth to be altered, including selecting a compound from a plurality of tooth colored compounds to match the shade of the adjacent or surrounding teeth in the individual's mouth;
- c) applying the covering compound prepared in step b) to the tooth surface to be coated;
- d) allowing the covering compound applied to the tooth to dry by exposing the tooth to air;
- e) wherein the step of preparing a tooth further includes the steps of providing a weak acid solution and contacting the tooth surface with the weak acid solution; and
- f) wherein the weak acid solution is a citric acid of lemon juice.

33. A composition for cosmetically altering the appearance of teeth to whiten or colorize the teeth by applying said composition to a tooth surface, said composition comprising:

- a) a lac resin;
- b) a solvent for dissolving said lac resin;
- c) a pigment;
- d) wherein said solvent comprises ethanol; and
- e) wherein said lac resin comprises refined bleached food grade dewaxed lac.

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L7: Entry 47 of 106

File: USPT

Jun 2, 1998

DOCUMENT-IDENTIFIER: US 5759251 A

TITLE: Titanium dioxide ceramic paint and methods of producing same

Brief Summary Text (37):

The peroxotitanic acid usable for the ceramic paint of the present invention can be prepared in the state of an aqueous solution by adding an aqueous hydrogen peroxide solution to an aqueous solution of orthotitanic acid prepared by the above-mentioned procedures.

Brief Summary Text (45):

Also, the peroxotitanic acid and titanium dioxide particle-containing aqueous paint of the present invention can be prepared by adding a peroxide compound, for example, hydrogen peroxide, into the aqueous orthotitanic salt solution, before or after the hydrolysis procedures. In this case, the content of peroxotitanic acid in the resultant aqueous paint can be adjusted to 0 to 100% by controlling the amount of the peroxide compound to be added to the aqueous titanium salt solution. Namely, when no peroxide compound is added, the resultant aqueous paint contains orthotitanic acid and is free from peroxotitanic acid. Also, when the peroxide compound is added in an amount equimolar or more to the molar amount of the orthotitanic acid, the resultant aqueous paint contains peroxotitanic acid and is free from orthotitanic acid.

Brief Summary Text (51):

When anhydrous titanium chloride is used as a starting material, an aqueous titanium chloride solution can be prepared by gradually dissolving anhydrous titanium chloride in pure water while cooling with ice. Also, when titanium trichloride is employed as a starting material, first, titanium (III) ions are oxidized into titanium (IV) ions with an oxidizing agent, for example, hydrogen peroxide, and second, the resultant titanium tetrachloride is subjected to the aqueous paint-producing procedures.

Brief Summary Text (98):

In the dispersion of the collected precipitate, the peroxide compound is preferably selected from hydrogen peroxide, sodium peroxide, and barium peroxide. The peroxide compound react with at least a portion of orthotitanic acid and titanium (IV) ions to produce peroxotitanic acid. When hydrogen peroxide is used, the addition of hydrogen peroxide can be carried out before the collection of the precipitate. Also when sodium peroxide or barium peroxide is used, the resultant aqueous paint may be further subjected to the above-mentioned treatment (a), (b) and/or (c) to remove sodium ions or barium ions from the aqueous paint.

Detailed Description Text (4):

In each of Examples 1 to 4, an aqueous paint was prepared by mixing an aqueous 10% sodium hydroxide solution to an aqueous 5% titanium tetrachloride solution to cause a precipitate to generate, collecting the precipitate consisting of orthotitanic acid by filtration, rinsing the collected precipitate with water, dispersing the precipitate in water, mixing the resultant orthotitanic acid-containing aqueous liquid with crystalline titanium dioxide particles, and then mixing the resultant liquid with the complexing agent in the amount as indicated in Table 1 in each of Examples 1, 3 and 4, and with the aqueous hydrogen peroxide solution in the amount sufficient to realize the concentration of peroxotitanic acid as shown in Table 1 in Example 2, and fully agitating the resultant mixture liquid by using a homomixer. The above-mentioned crystalline titanium dioxide particles were available under the

trademark of titanium dioxide P-25 (anatase or rutile) made from Nihon Aerosil K.K. The above-mentioned complexing agent consisted of acetylacetone in Example 1, lactic acid in Example 3 and oxalic acid in Example 4.

Detailed Description Text (7):

An aqueous 10% titanium tetrachloride solution was mixed with sodium hydroxide in an amount of 1 mole per mole of titanium present in the titanium tetrachloride solution, the mixed liquid was heated at a temperature of 90.degree. C. for 40 minutes, the heated liquid was mixed with an aqueous 5% sodium hydroxide solution in such an amount that the resultant mixed liquid became neutral, to cause a precipitate to form, the resultant precipitate comprising orthotitanic acid and titanium dioxide was dispersed in water, the resultant aqueous dispersion was mixed with an aqueous hydrogen peroxide solution in such an amount that the resultant mixed liquid had the concentration of peroxotitanic acid as shown in Table 1, the resultant mixture liquid was fully agitated by using a homomixer. As aqueous titanium dioxide ceramic paint was obtained.

Detailed Description Text (11):

An aqueous 10% titanium tetrachloride solution was mixed with sodium hydroxide in an amount of 2 moles per mole of titanium present in the titanium tetrachloride solution, the mixed liquid was heated at a temperature of 75.degree. C. for 20 minutes, the heated liquid was mixed with an aqueous 5% sodium hydroxide solution in such an amount that the resultant mixed liquid became neutral, to cause a precipitate to generate, the resultant precipitate comprising orthotitanic acid and titanium dioxide was dispersed in water, the resultant aqueous dispersion was mixed with, in Example 7, gluconic acid in the concentration shown in Table 1, and in Example 8, an aqueous hydrogen peroxide solution in such an amount that the resultant mixed liquid had the concentration of peroxotitanic acid as shown in Table 1, the resultant mixture liquid was fully agitated by using a homomixer. As aqueous titanium dioxide ceramic paint was obtained.

Detailed Description Text (20):

An aqueous 20% by weight titanium trichloride solution was placed in a beaker and diluted with water. The diluted solution was mixed with an aqueous 31% hydrogen peroxide solution until violet-colored titanium (III) ions present in the aqueous solution were completely converted to colorless titanium (IV) ions. The resultant aqueous titanium tetrachloride solution was mixed with an aqueous solution of 10% by weight of sodium hydroxide in an amount of 2 moles per mole of titanium present in the aqueous solution. The mixed liquid was heated at a temperature of 70.degree. C. for 30 minutes and then cooled with water to a temperature of 30.degree. C. The resultant liquid was subjected to a diffusion dialysis treatment using an RO membrane at a temperature of 40.degree. C. while passing deionized water through the membrane, to prepare an aqueous titanium dioxide ceramic paint substantially free from impurity ions.

Detailed Description Text (22):

An aqueous 20% by weight titanium trichloride solution was placed in a beaker and diluted with water. The diluted solution was mixed with an aqueous 31% hydrogen peroxide solution until violet-colored titanium (III) ions present in the aqueous solution were completely converted to colorless titanium (IV) ions. The resultant aqueous titanium tetrachloride solution was mixed with an aqueous solution of 10% by weight of sodium hydroxide in an amount of 0.24 mole per mole of titanium present in the aqueous solution. The mixed liquid was heated at a temperature of 95.degree. C. for 5 minutes and then cooled with water to a temperature of 30.degree. C. The resultant liquid was subjected to a diffusion dialysis treatment using anion exchange and cation exchange membranes at a temperature of 30.degree. C. while passing deionized water through the membrane, to prepare an aqueous titanium dioxide-containing liquid substantially free from impurity ions. The aqueous titanium dioxide-containing liquid was mixed with a complexing agent consisting of acetylacetone in an amount of 1.2% by weight.

Detailed Description Text (55):

In each of Comparative Examples 1 to 3, an aqueous titanium dioxide ceramic paint was prepared by mixing an aqueous solution of 5% by weight of titanium tetrachloride with an aqueous solution of 10% by weight of sodium hydroxide to generate a

precipitate, the precipitate was collected and rinsed with water, the resultant orthotitanic acid precipitate was dispersed in water, the resultant aqueous orthotitanic acid solution was mixed with titanium dioxide particles and then with an aqueous hydrogen peroxide solution in such an amount that the resultant solution contained peroxotitanic acid in the concentration as shown in Table 1, and the resultant mixture was fully agitated. The mixed titanium dioxide particles were, in Comparative Example 1, rutile type titanium dioxide pigment particles having an average particle size of 0.5 μm , in Comparative Example 2, titanium dioxide pigment particles (anatase or rutile) (trademark: P-25, made by Nihon Aerosil K.K.) and in Comparative Example 3, anatase type titanium dioxide colloidal particles having an average particle size of 0.01 μm and in the form of an aqueous colloidal solution, prepared by heating an aqueous solution of 8% by weight of titanium oxychloride at a temperature of 75.degree. C. for 20 minutes, and subjected to the heated solution to a diffusion dialysis treatment using an ion-exchange membrane.

Detailed Description Text (71):

A sample of an aqueous paint was filtered through a 5 C filter paper sheet, the resultant filtrate was acidified with hydrochloric acid and then mixed with hydrogen peroxide to convert orthotitanic acid and titanium (IV) ions to peroxotitanic acid. The absorbance of the resultant modified filtrate was measured at a wavelength of 430 nm by the same method as mentioned above. From the measurement result, the total concentration of orthotitanic acid and titanium (IV) ions in the sample was determined. When the sample contained peroxotitanic acid, the concentration of the peroxotitanic acid in the sample was determined by the method mentioned in the above item (2), and resultant peroxotitanic acid concentration is subtracted from the total concentration of orthotitanic acid and/or titanium (IV) ions and peroxotitanic acid determined above.

CLAIMS:

1. An aqueous titanium dioxide ceramic paint for forming a paint coating having excellent hydrophilicity, photocatalytic activity and light-transmittance, comprising:

(A) at least one member selected from the group consisting of orthotitanic acid, titanium (IV) ions and peroxotitanic acid, and

(B) crystalline titanium dioxide colloidal particles having an average particle size of 0.001 to 0.2 μm .

the weight ratio, in terms of titanium, of the component (A) to the component (B) being from 1:0.1 to 1:200, and the ceramic paint being substantially free from impurity ions.

2. The aqueous titanium dioxide ceramic paint as claimed in claim 1, wherein the average particle size of the crystalline titanium dioxide particles is from 0.002 to 0.1 μm .

3. A method of producing the aqueous titanium dioxide ceramic paint as claimed in claim 1, comprising subjecting an aqueous solution of a titanium salt to at least one treatment selected from the group consisting of:

(a) a dialysis treatment using a semipermeable membrane,

(b) an electrodialysis treatment using a semipermeable membrane, and

(c) an ion-exchange treatment using an ion-exchanger

to thereby hydrolyze and convert at least a portion of the titanium salt to be orthotitanic acid and the crystalline titanium dioxide colloidal particles, while removing impurity ions from the aqueous titanium salt solution.

8. A method of producing the titanium dioxide ceramic paint as claimed in claim 1, comprising the steps of:

mixing an aqueous solution of a titanium salt with at least one member selected from the group consisting of alkali metal hydroxides and ammonia in an amount of less than 4 moles per mole of titanium present in the aqueous titanium salt solution; and

heating the resultant mixed aqueous solution at a temperature of 50.degree. C. or more but less than 100.degree. C., to thereby hydrolyze and convert at least a portion of the titanium salt to the orthotitanic acid and the crystalline titanium dioxide colloidal particles; and

mixing the resultant titanium dioxide colloidal particle-containing aqueous solution with at least one member selected from the group consisting of alkali metal hydroxides and ammonia to cause a precipitate to be formed;

collecting the resultant precipitate from the aqueous solution; and

dispersing the collected precipitate in a dispersing medium consisting of a member selected from the group consisting of water, aqueous solutions of peroxide compounds and aqueous solutions of complexing agents for titanium .

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L7: Entry 47 of 106

File: USPT

Jun 2, 1998

US-PAT-NO: 5759251

DOCUMENT-IDENTIFIER: US 5759251 A

TITLE: Titanium dioxide ceramic paint and methods of producing same

DATE-ISSUED: June 2, 1998

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Nakamura; Mitsuru	Tokyo			JP
Mori; Kazuhiko	Tokyo			JP
Tanaka; Masanobu	Tokyo			JP
Nishizawa; Yoshihiko	Tokyo			JP

US-CL-CURRENT: 106/286.4; 106/287.19, 106/816

CLAIMS:

What we claimed is:

1. An aqueous titanium dioxide ceramic paint for forming a paint coating having excellent hydrophilicity, photocatalytic activity and light-transmittance, comprising:

(A) at least one member selected from the group consisting of orthotitanic acid, titanium (IV) ions and peroxotitanic acid, and

(B) crystalline titanium dioxide colloidal particles having an average particle size of 0.001 to 0.2 .mu.m.

the weight ratio, in terms of titanium, of the component (A) to the component (B) being from 1:0.1 to 1:200, and the ceramic paint being substantially free from impurity ions.

2. The aqueous titanium dioxide ceramic paint as claimed in claim 1, wherein the average particle size of the crystalline titanium dioxide particles is from 0.002 to 0.1 .mu.m.

3. A method of producing the aqueous titanium dioxide ceramic paint as claimed in claim 1, comprising subjecting an aqueous solution of a titanium salt to at least one treatment selected from the group consisting of:

(a) a dialysis treatment using a semipermeable membrane,

(b) an electrodialysis treatment using a semipermeable membrane, and

(c) an ion-exchange treatment using an ion-exchanger

to thereby hydrolyze and convert at least a portion of the titanium salt to be orthotitanic acid and the crystalline titanium dioxide colloidal particles, while removing impurity ions from the aqueous titanium salt solution.

4. The method as claimed in claim 3, wherein the treatments (a), (b) and (c) are carried out at a temperature of 0.degree. to 80.degree. C.

5. A method as claimed in claim 4, wherein the aqueous solution of the titanium salt is heated at a temperature of 50.degree. C. or more but less than 100.degree. C., and then, subjected to the at least one treatment selected from the treatments (a), (b) and (c).

6. The method as claimed in claim 3 or 5, wherein the aqueous titanium salt solution is an aqueous solution containing at least one member selected from the group consisting of titanium oxychloride and titanium oxysulfate.

7. The method as claimed in claim 3, wherein the aqueous titanium salt solution is mixed with at least one member selected from the group consisting of alkali metal hydroxides and ammonia in an amount of less than 4 moles per mole of titanium present in the aqueous titanium salt solution, and the resultant mixed aqueous solution is heated at a temperature of 50.degree. C. or more but less than 100.degree. C., and then subjected to the at least one treatment selected from the treatments (a), (b) and (c).

8. A method of producing the titanium dioxide ceramic paint as claimed in claim 1, comprising the steps of:

mixing an aqueous solution of a titanium salt with at least one member selected from the group consisting of alkali metal hydroxides and ammonia in an amount of less than 4 moles per mole of titanium present in the aqueous titanium salt solution; and

heating the resultant mixed aqueous solution at a temperature of 50.degree. C. or more but less than 100.degree. C., to thereby hydrolyze and convert at least a portion of the titanium salt to the orthotitanic acid and the crystalline titanium dioxide colloidal particles; and

mixing the resultant titanium dioxide colloidal particle-containing aqueous solution with at least one member selected from the group consisting of alkali metal hydroxides and ammonia to cause a precipitate to be formed;

collecting the resultant precipitate from the aqueous solution; and

dispersing the collected precipitate in a dispersing medium consisting of a member selected from the group consisting of water, aqueous solutions of peroxide compounds and aqueous solutions of complexing agents for titanium .

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L7: Entry 47 of 106

File: USPT

Jun 2, 1998

US-PAT-NO: 5759251

DOCUMENT-IDENTIFIER: US 5759251 A

TITLE: Titanium dioxide ceramic paint and methods of producing same

DATE-ISSUED: June 2, 1998

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
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Mori; Kazuhiko	Tokyo			JP
Tanaka; Masanobu	Tokyo			JP
Nishizawa; Yoshihiko	Tokyo			JP

ASSIGNEE-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY	TYPE CODE
Nihon Parkerizing Co., Ltd.	Tokyo			JP	03

APPL-NO: 08/ 863629 [PALM]

DATE FILED: May 27, 1997

FOREIGN-APPL-PRIORITY-DATA:

COUNTRY	APPL-NO	APPL-DATE
JP	8-130090	May 24, 1996
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INT-CL: [06] C09 D 1/00, C04 B 7/345

US-CL-ISSUED: 106/286.4; 106/287.19, 106/816

US-CL-CURRENT: 106/286.4; 106/287.19, 106/816

FIELD-OF-SEARCH: 106/286.4, 106/287.19, 106/816

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

Search Selected

Search ALL

PAT-NO	ISSUE-DATE	PATENTEE-NAME	US-CL
<input type="checkbox"/> 5403513	April 1995	Sato et al.	252/309

FOREIGN PATENT DOCUMENTS

FOREIGN-PAT-NO	PUBN-DATE	COUNTRY	US-CL
4-83537	March 1992	JP	106/286.4
4-142372	May 1992	JP	
6-293519	October 1994	JP	
7-100378	April 1995	JP	

ART-UNIT: 118

PRIMARY-EXAMINER: Brunsman; David

ABSTRACT:

A hydrophilic, photocatalytic, transparent and adhesive aqueous TiO_2 ceramic paint comprises orthotitanic acid, Ti^{4+} ions and/or peroxotitanic acid and crystalline TiO_2 colloidal particles with an average particle size of 0.001 to 0.2 μm , and is produced by applying (a) semipermeable membrane dialysis, (b) semipermeable membrane electrodialysis or (c) ion-exchange treatment to an aqueous Ti salt solution to thereby hydrolyze and convert at least a portion of the Ti salt to the orthotitanic acid and the TiO_2 colloidal particles and simultaneously to remove impurity ions from the aqueous solution, or by heating the Ti salt solution at 50.degree. to 100.degree. C. after or without mixing an alkali metal hydroxide or ammonia to the Ti salt solution, and then applying the treatment (a), (b) or (c) to the resultant solution, or by mixing an alkali metal hydroxide or ammonia to the heated Ti salt solution followed by collecting a resultant precipitate and dispersing the precipitate in water or an aqueous solution of a peroxide compound or complexing agent.

8 Claims, 0 Drawing figures

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L7: Entry 48 of 106

File: USPT

Dec 16, 1997

DOCUMENT-IDENTIFIER: US 5698205 A

TITLE: Photostabilization of titanium dioxide sols

Brief Summary Text (20):

Since the discoloration of the TiO.sub.2 sol by darkening is evidently based on the reduction of Ti.sup.4+ to Ti.sup.3+, this can also be suppressed by suitable oxidizing agents. Inorganic peroxides, such as, for example, hydrogen peroxide, sodium perchlorate, potassium perchlorate, perborates or peroxodisulfate, which are employed in an amount of 0.1-20 mole %, preferably 1-10 mole %, in particular 5-10 mole %, based on the TiO.sub.2 content of the sol, have proved to be particularly suitable.

CLAIMS:

1. A titanium dioxide sol comprising:titanium dioxide sol particles,

an aqueous dispersion medium,

5-40 mole %, based on TiO.sub.2 content of sol, of a hydroxycarboxylic acid or a salt, ester or amide thereof as a stabilizing agent, and

0.1-20 mole %, based on TiO.sub.2 content of sol, of a further stabilizing agent, wherein said further stabilizing agent is selected from the group consisting of La.sup.2+, Fe.sup.2+, Fe.sup.3+, Sn.sup.4+, Zn.sup.2+, Ce.sup.3+, Mn.sup.2+, inorganic fluorides, inorganic iodates, inorganic nitrates, one or more complexing agents, one or more oxidizing agents, and combinations thereof,

wherein said sol is photostable and neutral.

2. A photostable titanium dioxide sol according to claim 1, wherein said hydroxycarboxylic acid is citric acid or tartaric acid.3. A photostable titanium dioxide sol according to claim 1, wherein said further stabilizing agent is selected from the group consisting of La.sup.2+, Zn.sup.2+, Sn.sup.4+, Mn.sup.2+, Ce.sup.3+, Fe.sup.2+ and Fe.sup.3+.4. A photostable titanium dioxide sol according to claim 1, wherein said further stabilizing agent is selected from the group consisting of [F.sup.-, IO.sup.-.sub.3, SO.sub.4.sup.2+ and NO.sub.3.sup.2-] inorganic fluorides, inorganic iodates and inorganic nitrates.5. A photostable titanium dioxide sol according to claim 1, wherein said further stabilizing agent is EDTA.6. A photostable titanium dioxide sol according to claim 1, wherein said further stabilizing agent is a perchlorate or a peroxodisulfate.7. A photostable titanium dioxide sol according to claim 1, wherein said hydroxycarboxylic acid is citric acid or tartaric acid; said complexing agent is EDTA; and said oxidizing agents are perchlorates and peroxodisulfates.

8. A titanium dioxide sol according to claim 1, wherein said oxidizing agents are hydrogen peroxide, perchlorates, perborates and peroxodisulfates.

9. A process for the preparation of a photostable titanium dioxide sol, said sol comprising titanium dioxide sol particles, and an aqueous dispersion medium, said process comprising:

stabilizing said sol by adding 5-40 mole %, based on TiO_2 content of said sol, of a hydroxycarboxylic acid or a salt, ester or amide thereof as a stabilizing agent, and

after stabilization of said sol with said hydroxycarboxylic acid or a salt, ester or amide thereof, said sol having a pH of 6-8, adding 0.1-20 mole %, based on TiO_2 content of said sol, of a further stabilizing agent selected from the group consisting of metal ions, anions, one or more complexing agents, one or more oxidizing agents, and combinations thereof.

10. A process for preparing a dried photostable titanium dioxide sol, comprising:

drying a sol prepared according to claim 9, and further comprising drying said sol.

12. A process for precipitating a photostable titanium dioxide sol comprising: adding an alcohol to a sol prepared according to claim 9.

13. In a cosmetic formulation containing a carrier and a titanium dioxide sol particle, the improvement wherein said formulation contains a titanium dioxide sol prepared according to claim 9.

16. A process according to claim 9, wherein said oxidizing agents are hydrogen peroxide, perchlorates, perborates and peroxodisulfates.

17. A titanium dioxide sol comprising:

titanium dioxide sol particles having a particle size of 5-200 nm,

an aqueous dispersion medium,

5-40 mole %, based on TiO_2 content of sol, of a stabilizing agent selected from the group consisting of hydroxy carboxylic acid having 1-6 hydroxy groups and 1-4--COOH groups, a monoalkyl amide thereof wherein the alkyl group has 1-10 C atoms, a dialkyl amide thereof wherein each, alkyl group has 1-10 C atoms, an alkali metal salt thereof, an alkaline earth salt thereof, and an amine salt thereof, and

0.1-20 mole %, based on TiO_2 content of sol, of a further stabilizing agent, wherein said further stabilizing agent is at least one metal ion, at least one anion, at least one complexing agent, at least one oxidizing agent, or combinations thereof,

wherein said at least one metal ion is La^{2+} , Zn^{2+} , Sn^{4+} , Mn^{2+} , Ce^{3+} , Fe^{2+} , Fe^{3+} , or combinations thereof,

wherein said at least one anion are an inorganic fluoride, an inorganic iodate, an inorganic nitrate or combinations thereof,

wherein said at least one complexing agent is EDTA, nitrilotriacetic acid, 1,2-di-aminocyclohexylene-dinitrilotetraacetic acid, diethylenetriaminepentaacetic acid, N-(2-hydroxy-ethyl)-ethylenediamino-N,N,N-triacetic acid, tetraethylenetetraaminohexaacetic acid or combinations thereof, and

wherein said at least one oxidizing agent is an inorganic peroxide or combinations thereof.

18. A titanium dioxide sol according to claim 17, wherein said at least one oxidizing agent is hydrogen peroxide, a perchlorate, a perborate, a peroxodisulfate or combinations thereof.

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L7: Entry 48 of 106

File: USPT

Dec 16, 1997

US-PAT-NO: 5698205

DOCUMENT-IDENTIFIER: US 5698205 A

TITLE: Photostabilization of titanium dioxide sols

DATE-ISSUED: December 16, 1997

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
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Hamann; Ulrike	Darmstadt			DE
Heyland; Andrea	Reichelsheim			DE

US-CL-CURRENT: 424/401; 252/363.5, 423/610, 423/612, 424/59, 424/70.9, 516/90,
516/905, 516/918, 516/919

CLAIMS:

What is claimed is:

1. A titanium dioxide sol comprising:titanium dioxide sol particles,

an aqueous dispersion medium,

5-40 mole %, based on TiO_2 content of sol, of a hydroxycarboxylic acid or a salt, ester or amide thereof as a stabilizing agent, and0.1-20 mole %, based on TiO_2 content of sol, of a further stabilizing agent, wherein said further stabilizing agent is selected from the group consisting of La^{2+} , Fe^{2+} , Fe^{3+} , Sn^{4+} , Zn^{2+} , Ce^{3+} , Mn^{2+} , inorganic fluorides, inorganic iodates, inorganic nitrates, one or more complexing agents, one or more oxidizing agents, and combinations thereof,

wherein said sol is photostable and neutral.

2. A photostable titanium dioxide sol according to claim 1, wherein said hydroxycarboxylic acid is citric acid or tartaric acid.3. A photostable titanium dioxide sol according to claim 1, wherein said further stabilizing agent is selected from the group consisting of La^{2+} , Zn^{2+} , Sn^{4+} , Mn^{2+} , Ce^{3+} , Fe^{2+} and Fe^{3+} .4. A photostable titanium dioxide sol according to claim 1, wherein said further stabilizing agent is selected from the group consisting of $[\text{F}^{sup.-}, \text{IO}^{sup.-.sub.3}, \text{SO}^{sub.4.sup.2+}$ and $\text{NO}^{sub.3.sup.2-}]$ inorganic fluorides, inorganic iodates and inorganic nitrates.5. A photostable titanium dioxide sol according to claim 1, wherein said further stabilizing agent is EDTA.6. A photostable titanium dioxide sol according to claim 1, wherein said further stabilizing agent is a perchlorate or a peroxodisulfate.

7. A photostable titanium dioxide sol according to claim 1, wherein said hydroxycarboxylic acid is citric acid or tartaric acid; said complexing agent is EDTA; and said oxidizing agents are perchlorates and peroxodisulfates.

8. A titanium dioxide sol according to claim 1, wherein said oxidizing agents are hydrogen peroxide, perchlorates, perborates and peroxodisulfates.

9. A process for the preparation of a photostable titanium dioxide sol, said sol comprising titanium dioxide sol particles, and an aqueous dispersion medium, said process comprising:

stabilizing said sol by adding 5-40 mole %, based on TiO_2 content of said sol, of a hydroxycarboxylic acid or a salt, ester or amide thereof as a stabilizing agent, and

after stabilization of said sol with said hydroxycarboxylic acid or a salt, ester or amide thereof, said sol having a pH of 6-8, adding 0.1-20 mole %, based on TiO_2 content of said sol, of a further stabilizing agent selected from the group consisting of metal ions, anions, one or more complexing agents, one or more oxidizing agents, and combinations thereof.

10. A process for preparing a dried photostable titanium dioxide sol, comprising:

drying a sol prepared according to claim 9, and further comprising drying said sol.

11. A process according to claim 10, further comprising precipitating said sol with an alcohol prior to drying.

12. A process for precipitating a photostable titanium dioxide sol comprising: adding an alcohol to a sol prepared according to claim 9.

13. In a cosmetic formulation containing a carrier and a titanium dioxide sol particle, the improvement wherein said formulation contains a titanium dioxide sol prepared according to claim 9.

14. A process according to claim 9, wherein said metal ions are La^{2+} , Fe^{2+} , Fe^{3+} , Sn^{4+} , Zn^{2+} , Ce^{3+} and Mn^{2+} , and said anions are inorganic fluorides, inorganic iodates and inorganic nitrates.

15. A process according to claim 14, wherein said one or more complexing agents are EDTA, nitrilotriacetic acid, 1,2-diaminocyclohexylene-dinitrilotetraacetic acid, diethylenetriamine pentaacetic acid, N-(2-hydroxy-ethyl)-ethylenediamino-N,N,N-triacetic acid and tetraethylenetetraaminohexaacetic acid, and said one or more oxidizing agents are perchlorates and peroxodisulfates.

16. A process according to claim 9, wherein said oxidizing agents are hydrogen peroxide, perchlorates, perborates and peroxodisulfates.

17. A titanium dioxide sol comprising:

titanium dioxide sol particles having a particle size of 5-200 nm,

an aqueous dispersion medium,

5-40 mole %, based on TiO_2 content of sol, of a stabilizing agent selected from the group consisting of hydroxy carboxylic acid having 1-6 hydroxy groups and 1-4--COOH groups, a monoalkyl amide thereof wherein the alkyl group has 1-10 C atoms, a dialkyl amide thereof wherein each, alkyl group has 1-10 C atoms, an alkali metal salt thereof, an alkaline earth salt thereof, and an amine salt thereof, and

0.1-20 mole %, based on TiO_2 content of sol, of a further stabilizing agent, wherein said further stabilizing agent is at least one metal ion, at least one anion, at least one complexing agent, at least one oxidizing agent, or combinations thereof,

wherein said at least one metal ion is La^{2+} , Zn^{2+} , Sn^{4+} , Mn^{2+} , Ce^{3+} , Fe^{2+} , Fe^{3+} , or combinations thereof,

wherein said at least one anion are an inorganic fluoride, an inorganic iodate, an inorganic nitrate or combinations thereof,

wherein said at least one complexing agent is EDTA, nitrilotriacetic acid, 1,2-di-aminocyclohexylene-dinitrilotetraacetic acid, diethylenetriaminepentaacetic acid, N-(2-hydroxy-ethyl)-ethylenediamino-N,N,N-triacetic acid, tetraethylenetetraaminohexaacetic acid or combinations thereof, and

wherein said at least one oxidizing agent is an inorganic peroxide or combinations thereof.

18. A titanium dioxide sol according to claim 17, wherein said at least one oxidizing agent is hydrogen peroxide, a perchlorate, a perborate, a peroxodisulfate or combinations thereof.

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L7: Entry 49 of 106

File: USPT

Nov 18, 1997

US-PAT-NO: 5688492

DOCUMENT-IDENTIFIER: US 5688492 A

TITLE: Oral hygiene composition

DATE-ISSUED: November 18, 1997

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Galley; Edward	Nottingham			GB2
Cooper; Michael David	Nottingham			GB2

US-CL-CURRENT: 424/49; 424/421, 424/440, 424/464, 424/468, 424/48, 424/489, 424/52,
424/53, 424/54, 424/600, 424/604, 424/606, 424/617, 424/618, 424/630, 424/635,
424/641, 424/642, 424/643, 424/649, 424/650, 424/688, 424/691

CLAIMS:

We claim:

1. An oral composition suitable for use in oral hygiene or dental treatment comprising an effective amount of an antimicrobial agent in the form of a particulate, non-ion-exchanging, zeolite-free, inert metal oxide, a sparingly soluble metal salt providing antimicrobial metal ions in use and a further material selected from the group consisting of humectants, gelling agents, abrasives, fluoride sources, desensitizing agents, flavorings, colorings, sweeteners, preservatives, structuring agents, bactericides, anti-tartar agents and anti-plaque agents.
2. A composition as claimed in claim 1, wherein the metal salt is selected from the group consisting of silver, copper, gold, platinum and zinc salts.
3. A composition as claimed in claim 1, wherein the metal oxide is selected from the group consisting of titanium dioxide, aluminum oxide, zirconium oxide, hafnium oxide, niobium oxide, tantalum oxide, calcium hydroxyapatite, barium sulphate, and magnesium oxide.
4. A composition as claimed in claim 3, wherein the titanium dioxide is in at least one of anatase, rutile and brookite crystalline form.
5. A composition as claimed in claim 1 wherein the antimicrobial agent is employed in combination with a further antimicrobial agent providing short-term antimicrobial action.
6. A composition as claimed in claim 1 wherein the metal salt is present in an amount of from about 0.5 to 75% by weight of the antimicrobial agent.
7. A composition as claimed in claim 6 wherein the metal salt is present in an amount of from about 1 to 50% by weight of the antimicrobial agent.
8. A composition as claimed in claim 7 wherein the metal salt is present in an amount of from about 2 to 25% by weight of the antimicrobial agent.
9. A composition as claimed in claim 1 wherein the metal oxide has an average

particle size of less than about 25 micrometers.

10. A composition as claimed in claim 9 wherein the average particle size is between about 0.01 and 15 micrometers.

11. A composition as claimed in claim 1 wherein the metal oxide is titanium dioxide having a primary crystal size of from about 0.005 to 5 micrometers.

12. A composition as claimed in claim 11 wherein the titanium dioxide has a primary crystal size of from about 0.01 to 1 micrometers.

13. A composition as claimed in claim 1 wherein the metal oxide has a mean surface area of from about 1 to 300 m.² /g.

14. A composition as claimed in claim 1 wherein the antimicrobial agent is included in the composition in an amount of from about 1.times.10.⁻⁵ to 5% by weight.

15. A composition as claimed in claim 14 wherein the antimicrobial agent is included in the composition in an amount of from about 0.01% to 3% by weight.

16. A method of cleaning a mouth of an individual for cosmetic purposes, comprising applying to the mouth an oral hygiene composition comprising an effective amount of an antimicrobial agent comprising a particulate, non-ion-exchanging, zeolite-free, inert metal oxide and a sparingly soluble metal salt providing antimicrobial metal ions in use.

17. A method of treating or preventing dental caries or gingivitis, comprising applying to a mouth of an individual an oral hygiene composition comprising an effective amount of an antimicrobial agent comprising a particulate, non-ion-exchanging, zeolite-free, inert metal oxide and a sparingly soluble metal salt providing antimicrobial metal ions in use.

18. A method of providing sustained antimicrobial activity in a mouth of an individual, comprising applying to the mouth an oral hygiene composition comprising an effective amount of an antimicrobial agent comprising a particulate, non-ion-exchanging, zeolite-free, inert metal oxide and a sparingly soluble metal salt providing antimicrobial metal ions in use.

19. A composition as claimed in claim 5, wherein the further antimicrobial agent is selected from the group consisting of a zinc salt, a tin salt, cetyl pyridinium chloride, a bis-biguanide, aliphatic amines, bromochlorophene, hexachlorophene, salicylanilides, a quaternary ammonium compound and triclosan.

20. A composition as claimed in claim 1, wherein the composition is selected from the group consisting of a dentrifice, mouthwash, tooth powder, chewing gum, lozenge, mouth spray, denture cleansing formulation, tooth paint and glass ionomer cement.

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L7: Entry 49 of 106

File: USPT

Nov 18, 1997

DOCUMENT-IDENTIFIER: US 5688492 A

TITLE: Oral hygiene composition

Brief Summary Text (1):

The present invention relates to an oral hygiene composition providing sustained antimicrobial action. There is also provided inter alia the use of such a composition in the treatment or prevention of dental caries or gingivitis and in the cleansing of the mouth for cosmetic purposes.

Brief Summary Text (2):

The term `oral hygiene composition` as used herein indicates a formulation for use in any form of oral hygiene or dental treatment and includes inter alia dentifrices, mouthwashes, toothpowders, chewing gums, lozenges, mouthsprays, flosses, denture cleansing formulations, tooth paints and glass ionomer cements.

Brief Summary Text (3):

The use of antimicrobial agents in oral hygiene compositions is well-known in the art. Typically, the anti-microbial agent is an organic species such as chlorhexidine, bromochlorophene, cetyl pyridinium chloride or benzethonium chloride. These agents are known to be effective against the microorganisms associated with dental caries and gingivitis, such as the bacteria Streptococcus mutans and Streptococcus salivarius, and the fungus Candida albicans.

Brief Summary Text (6):

The present invention seeks to solve this problem by providing an oral composition comprising an antimicrobial agent with a tendency to remain in the mouth in use, thus giving sustained antimicrobial activity and enhanced protection against dental caries and gingivitis.

Brief Summary Text (8):

JP-A-02283312 (Sanpo Seijaku KK) discloses a toothbrush having brush-hairs incorporating a bactericidal metal-carrying zeolite, the metal being carried on the zeolite by ion-exchange. The toothbrush is used for brushing teeth and gums.

Brief Summary Text (10):

It has now surprisingly been found that antimicrobial agents of the type disclosed in EP-A-0251783 are particularly well-suited to use in oral hygiene compositions. The agents in question show a high degree of substantivity to the mouth, and to the teeth and gums in particular, thus providing sustained oral antimicrobial action.

Brief Summary Text (20):

The oral composition may be formulated for use in any form of interdental or periodontal treatment and may be in the form, for example, of a dentifrice, mouthwash, toothpowder, chewing gum, lozenge, mouthspray, floss, dental paint, or glass ionomer cement. Use of the antimicrobial material of the present invention in a glass ionomer cement has the advantage of providing X-ray opacity as well as antimicrobial action.

Brief Summary Text (29):

Structuring agents may be required in, for example, dentifrices and gums to provide desirable textural properties and "mouthfeel". Suitable agents include natural gum binders such as gum tragacanth, xanthan gum, gum karaya and gum arabic, seaweed derivatives such as Irish moss and alginates, smectite clays such as bentonite or

hectorite, carboxyvinyl polymers and water-soluble cellulose derivatives such as hydroxyethyl cellulose and sodium carboxymethyl cellulose. Improved texture may also be achieved, for example, by including colloidal magnesium aluminium silicate. Suitably, the structuring agent is included in an amount of from 0-5%, preferably 0-3% by weight of the oral hygiene composition.

Brief Summary Text (30):

Abrasives should preferably be capable of cleaning and/or polishing the teeth without causing harm to dental enamel or dentine. They are used most commonly in dentifrices and toothpowders, but may also be used in mouthwashes etc. Suitable abrasives include the silica abrasives, such as hydrated silicas and silica gels, particularly silica xerogels such as those available under the trade name `Syloid` from W. R. Grace and Company. Also suitable are precipitated silica materials such as those available under the trade name `Zeodent` from J. M. Huber Corporation, and diatomaceous earths such as those available under the trade name `Celite` from Johns-Manville Corporation. Alternative abrasives include alumina, insoluble metaphosphates such as insoluble sodium metaphosphate, calcium carbonate, dicalcium phosphate (in dihydrate and anhydrous forms), calcium pyrophosphate (including .beta.-phase calcium) polymethoxylates and particulate thermosetting polymerised resins such as, for example, melamine-ureas, melamine-formaldehydes, urea-formaldehydes, melamine-urea-formaldehydes, cross-linked epoxides, melamines, phenolics, highly purified celluloses such as those available under the trade name `Elcema` from Degussa AG, and cross-linked polyesters. Suitably, abrasives are included in an amount of from 0-80%, preferably 0-60% by weight of the oral hygiene composition.

Brief Summary Text (32):

Preferably, the fluoride source is present in an amount sufficient to provide from about 50 ppm to about 4,000 ppm fluoride ions in use. Inclusion of a fluoride source is beneficial, since fluoride ions are known to become incorporated into the hydroxyapatite of tooth enamel, thereby increasing the resistance of the enamel to decay. Fluoride is also now thought to act locally on the tooth enamel, altering the remineralisation-demineralisation balance in favour of remineralisation. Inclusion of a fluoride source is also desirable when a polyphosphate anti-calculus agent is included, in order to inhibit the enzymic hydrolysis of such polyphosphates by salivary phosphatase enzymes.

Brief Summary Text (35):

Suitably, as described above, the compositions of the invention may include a further antimicrobial agent as a preservative and/or anti-plaque agent. Suitable antimicrobial agents include zinc salts (such as zinc citrate), cetyl pyridinium chloride, the bis-biguanides (such as chlorhexidine), aliphatic amines, bromochlorophene, hexachlorophene, salicylanilides, quaternary ammonium compounds and triclosan. Enzymic systems providing a source of a natural biocide may be used as alternatives to or in combination with the biocides listed. For example, a system comprising lactoperoxidase and glucose oxidase may be used to generate antimicrobial amounts of hydrogen peroxide in the presence of glucose, water and oxygen.

Brief Summary Text (40):

In a further aspect, the invention provides a method of treating or preventing dental caries or gingivitis by oral application of any oral hygiene composition as defined above.

Brief Summary Text (41):

In a further aspect, there is provided the use of any anti-microbial agent as defined above in the treatment or prevention of dental caries or gingivitis.

Brief Summary Text (42):

In a still further aspect, there is provided the use of any anti-microbial agent as defined above in the manufacture of a medicament for the treatment or prevention of dental caries or gingivitis.

Detailed Description Text (6):

An antimicrobial agent prepared substantially as described in Example 1 was tested for antimicrobial activity against Streptococcus salivarius and Streptococcus

mutans, which are organisms known to be associated with dental caries/gingivitis and against *Candida albicans*.

Detailed Description Text (10):

These results show the activity of the antimicrobial agent of the present invention against organisms known to be associated with dental caries/gingivitis. It will be noted that activity was not detected in the control.

Detailed Description Text (13):

The anti-plaque activity of the compositions according to the present invention has been demonstrated as follows. Thin strips of aluminium were used as "artificial tooth" surfaces on which plaque, collected from a small number of donors, was grown. Growth was encouraged by the provision of conditions resembling a normal oral environment (saliva, nutrients, pH and temperature) over a two day period with simulations made of the intake of two meals and of a sleeping, low nutrient period. The aluminium strips (and plaque) were exposed for one minute on two occasions to a suspension of the composition to be tested with distilled water and fresh saliva, thus simulating evening and morning toothbrushing sessions. Following the second exposure, plaque remaining on the strips after a four hour growth period and subsequent rinsing was dispersed by ultrasonic vibration. The optical density of the resulting plaque suspensions at 570 nm (two replicate readings per strip) was used to estimate the percentage reduction in plaque growth compared to plaque growth on test strips exposed to a control composition of saliva and water.

Detailed Description Text (34):

Antimicrobial tooth paint base

Detailed Description Text (35):

A tooth paint base is prepared in conventional manner to the following composition:

CLAIMS:

1. An oral composition suitable for use in oral hygiene or dental treatment comprising an effective amount of an antimicrobial agent in the form of a particulate, non-ion-exchanging, zeolite-free, inert metal oxide, a sparingly soluble metal salt providing antimicrobial metal ions in use and a further material selected from the group consisting of humectants, gelling agents, abrasives, fluoride sources, desensitizing agents, flavorings, colorings, sweeteners, preservatives, structuring agents, bactericides, anti-tartar agents and anti-plaque agents.
3. A composition as claimed in claim 1, wherein the metal oxide is selected from the group consisting of titanium dioxide, aluminum oxide, zirconium oxide, hafnium oxide, niobium oxide, tantalum oxide, calcium hydroxyapatite, barium sulphate, and magnesium oxide..
4. A composition as claimed in claim 3, wherein the titanium dioxide is in at least one of anatase, rutile and brookite crystalline form.
11. A composition as claimed in claim 1 wherein the metal oxide is titanium dioxide having a primary crystal size of from about 0.005 to 5 micrometers.
12. A composition as claimed in claim 11 wherein the titanium dioxide has a primary crystal size of from about 0.01 to 1 micrometers.
17. A method of treating or preventing dental caries or gingivitis, comprising applying to a mouth of an individual an oral hygiene composition comprising an effective amount of an antimicrobial agent comprising a particulate, non-ion-exchanging, zeolite-free, inert metal oxide and a sparingly soluble metal salt providing antimicrobial metal ions in use.
20. A composition as claimed in claim 1, wherein the composition is selected from the group consisting of a dentrifice, mouthwash, tooth powder, chewing gum, lozenge, mouth spray, denture cleansing formulation, tooth paint and glass ionomer cement.

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L7: Entry 54 of 106

File: USPT

Jul 30, 1996

US-PAT-NO: 5540907

DOCUMENT-IDENTIFIER: US 5540907 A

TITLE: Process for preparing high solids content titanium dioxide suspension with a dechlorinating agent

DATE-ISSUED: July 30, 1996

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Blumel; Siegfried	Ratingen			DE
Hartmann; Achim	Pulheim			DE
Thumm; Hans	Leverkusen			DE
Luginsland; Hans-Hermann	Leverkusen			DE
Rosendahl; Franz	Leverkusen			DE

US-CL-CURRENT: 423/610; 106/436

CLAIMS:

What is claimed is as follows:

1. A process for preparation of a titanium dioxide aqueous suspension, comprising the steps of

(a) dispensing titanium dioxide in an aqueous liquid to form a slurry having a maximum pH of about 4;

(b) reducing chlorine and reducible chlorine compounds in the slurry by adding a dechlorinating agent selected from the group consisting of at least one of hydroxyl ammonium chloride, hydrogen peroxide and sodium hydrogen sulfite in an amount which controls the multivalent anion content of the slurry sufficient to obtain, after the slurry is filtered in step (c), one of (i) a rigid filter cake with a solids content below about 60% by weight by controlling the content of multivalent anions in the slurry to about 5×10^{-3} mol/liter or (ii) a thixotropic filter cake with a solids content above about 65% by weight by controlling the content of the multivalent anions in the slurry to less than about 3×10^{-3} mol/liter; (c) filtering the slurry from step (b) to form a filter cake; and (d) thereafter rendering the filter cake flowable to form the titanium dioxide suspension.

2. The process according to claim 1, wherein step (b) includes a dechlorinating agent which introduces multivalent cations into the slurry.

3. The process according to claim 1, wherein the step of filtering comprises a pressure filtration apparatus.

4. The process according to claim 1, wherein the filter cake is rendered flowable by shearing.

5. The process according to claim 1, wherein the filtering step comprises a vacuum filtration on a Moore filter.

6. The process according to claim 1, wherein the dechlorinating agent is a

combination of hydrogen peroxide and sodium hydrogen sulfite.

7. The process according to claim 6, wherein addition of the hydrogen peroxide takes place before addition of sodium hydrogen sulfite.

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L7: Entry 54 of 106

File: USPT

Jul 30, 1996

DOCUMENT-IDENTIFIER: US 5540907 A

TITLE: Process for preparing high solids content titanium dioxide suspension with a dechlorinating agent

Abstract Text (1):

In the preparation of a suspension with a high content of titanium dioxide, which has been produced according to the chloride process, a finely divided titanium dioxide is slurried, in unflocculated condition without use of a dispersing agent, dechlorinated and then filtered. The content of multivalent anions can be controlled in the slurry before the filtration through suitable dechlorinating agents, particularly hydrogen peroxide and/or sodium hydrogen sulfite, and thereby the solid content in the filter cake and the consistency of the filter cake are advantageously influenced. If, on one hand, no or only few multivalent anions are present, a thixotropic filter cake with a high solids content is obtained in a pressure filtration. The filter cake is converted into a titanium dioxide suspension with a high solids content through feeding into a titanium dioxide suspension to which a dispersing agent has been added. If, on the other hand, a rigid filter cake is desired by, for example, a vacuum filtration, a minimum quantity of multivalent anions is necessary for the formation of the rigid filter cake. The solids content is, however, lowered in comparison to the thixotropic filter pastes. Where this minimum quantity of multivalent anions is exceeded, the solids content declines further. The rigid filter cakes can be rendered flowable through the direct admixing of dispersing agent. Hydrogen peroxide is particularly preferred as a dechlorinating agent.

Brief Summary Text (10):

In conventional processes for manufacturing titanium dioxide, the multivalent anions are largely introduced into the slurry by the dechlorinating agent. It is, therefore, especially preferred according to the present invention to use a dechlorinating agent which does not increase the content of multivalent anions in the suspension. A suitable substance has been found to be hydroxylammonium chloride and especially preferred is hydrogen peroxide.

Brief Summary Text (12):

According to a specific embodiment of the process of the present invention, a combination of hydrogen peroxide and sodium hydrogen sulfite with use of Moore filtering has proved effective. If hydrogen peroxide as well as sodium hydrogen sulfite are used as the dechlorinating agents, preferably the hydrogen peroxide addition should take place before the addition of sodium hydrogen sulfite.

Detailed Description Text (5):

Corresponding to the data given in Table 1 below showing Experiments 1 through 9 the slurry was completely or partially dechlorinated with sodium hydrogen sulfite solution and/or a corresponding quantity of 30% hydrogen peroxide solution. The completely or partially or undechlorinated slurry was filtered using laboratory suction filters. The consistency and the solids content of the filter cake obtained are reported in Table 1.

Detailed Description Text (10):

Partial quantities were withdrawn from a slurry with a titanium dioxide content of 510 g/l and a dechlorination requirement, calculated as equivalence concentration C (1/2 Cl.sub.2), of 10.times.10.sup.=3 mol/l and the dechlorination requirement increased through introduction of chlorine gas. (See Table 3 for the results

obtained). The pH in all experiments was at or below 4. All suspensions are dechlorinated with the same "base requirement" of the slurry not further chlorinated. The increased dechlorination requirement was leveled out before the addition of sodium hydrogen sulfite through the appropriate quantities of hydrogen peroxide. Irrespective of the dechlorination requirement, a filter paste with identical solids content was obtained. All filter cakes were rigid and were dispersed without problems through the direct addition of a dispersing agent (polyacrylate) and proved satisfactory.

Detailed Description Text (12):

This example involved a slurry with a titanium dioxide content of 564 g/l, a pH of 3.8 and a dechlorination requirement, calculated as equivalence concentration $C(1/2 Cl.sub.2) = 14 \times 10^{-3} \text{ mol/l}$ hydrogen peroxide and $5 \times 10^{-3} \text{ mol/l}$ sodium hydrogen sulfite and filtered on a Moore filtration apparatus. The filter paste had a solids content of 65%. A part of the paste was made flowable with 0.2% polyacrylate dispersing agent and 0.2% 2-amino-2-methylpropanol and spray dried (% details are based on $TiO.sub.2$). Subsequently the filter paste was concentrated to a solids content of 72% titanium dioxide with the dry material while the filter paste was produced and as much dispersing agent been added that a content of polyacrylate dispersing agent of 0.4% and 2-amino-2methylpropanol of 0.2% in the concentrated solution ensued (% data-active material based on $TiO.sub.2$). The suspension was ground in a 5 l sand mill with a capacity of 25 l/h; the viscosity of the suspension amounted to about 400 mPa.s.

CLAIMS:

1. A process for preparation of a titanium dioxide aqueous suspension, comprising the steps of

(a) dispensing titanium dioxide in an aqueous liquid to form a slurry having a maximum pH of about 4;

(b) reducing chlorine and reducible chlorine compounds in the slurry by adding a dechlorinating agent selected from the group consisting of at least one of hydroxyl ammonium chloride, hydrogen peroxide and sodium hydrogen sulfite in an amount which controls the multivalent anion content of the slurry sufficient to obtain, after the slurry is filtered in step (c), one of (i) a rigid filter cake with a solids content below about 60% by weight by controlling the content of multivalent anions in the slurry to about $5 \times 10^{-3} \text{ mol/liter}$ or (ii) a thixotropic filter cake with a solids content above about 65% by weight by controlling the content of the multivalent anions in the slurry to less than about $3 \times 10^{-3} \text{ mol/liter}$; (c) filtering the slurry from step (b) to form a filter cake; and (d) thereafter rendering the filter cake flowable to form the titanium dioxide suspension.

6. The process according to claim 1, wherein the dechlorinating agent is a combination of hydrogen peroxide and sodium hydrogen sulfite.

7. The process according to claim 6, wherein addition of the hydrogen peroxide takes place before addition of sodium hydrogen sulfite.

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L7: Entry 61 of 106

File: USPT

Mar 28, 1995

US-PAT-NO: 5401495

DOCUMENT-IDENTIFIER: US 5401495 A

TITLE: Teeth whitener

DATE-ISSUED: March 28, 1995

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Murayama; Ron	Laguna Niguel	CA		

US-CL-CURRENT: 424/49; 424/53

CLAIMS:

Having thus described the invention, I claim:

1. A three component system for whitening human teeth wherein each component is applied to the teeth in a sequential manner, said three component system comprising:

a first component comprising a conditioning mouth rinse capable of cleansing the surface of said teeth;

a second component comprising a viscous bleaching gel including an effective concentration of hydrogen peroxide for bleaching said teeth, said hydrogen peroxide present in an amount ranging from 1 to 10% by weight of the total gel; and

a third component comprising a viscous polishing composition including an abrasive substance for polishing said teeth and a pigmenting agent capable of imparting a white color to said teeth, whereby said conditioning rinse, said bleaching gel and said polishing composition are applied individually and seriatim to said teeth.

2. The system as set forth in claim 1 wherein said conditioning rinse comprises a dilute aqueous solution of acetic acid.

3. The system as set forth in claim 2 wherein said acetic acid is present in said rinse at a concentration of approximately 1.5 percent.

4. The system as set forth in claim 1 wherein said conditioning rinse further includes substances selected from the following: flavor enhancers, preservatives, surfactants and mixtures thereof.

5. The system as set forth in claim 4 wherein said flavor enhancers are selected from the group consisting of sweeteners and flavoring chemicals, said sweeteners are selected from the group consisting of saccharin, sodium saccharin, methyl salicylate, sorbitol and aspartame, said flavoring chemicals are selected from the group consisting of chemicals imparting the flavor of menthol, spearmint, wintergreen and cinnamon, said preservatives selected from the group consisting of sodium benzoate, and said surfactants are selected from the group consisting of polyoxyethylene (20) sorbitan monolaurate, sodium dodecyl sulfate, sodium lauryl sulfate and mixtures thereof.

6. The system as set forth in claim 1 wherein said effective concentration of said hydrogen peroxide in said gel is between 3 percent and 8 percent.
7. The system as set forth in claim 6 wherein said concentration of said hydrogen peroxide is 6 percent.
8. The system as set forth in claim 1 wherein said bleaching gel additionally comprises a gelling agent, a thickening agent, and a neutralizing agent.
9. The system as set forth in claim 8 wherein said gelling agent is selected from the group consisting of a copolymer of acrylic acid cross-linked with polyallyl sucrose, organic polymer acid colloids selected from the group consisting of polyuronic acids, carboxypolymethylene compounds, polyester resins containing three carboxyl groups, partially hydrolyzed polyacrylates, polymethacrylates, polyoxyethylenes, polypropylene copolymers and mixtures thereof.
10. The system as set forth in claim 9 wherein said gelling agent is a copolymer of acrylic acid cross-linked with approximately 0.75 percent to 1.5 percent polyallyl sucrose.
11. The system as set forth in claim 8 wherein said neutralizing agent is selected from the group consisting of sodium hydroxide, potassium hydroxide and triethanolamine.
12. The system as set forth in claim 8 wherein said thickening agent comprises non-ionic cellulose gums selected from the group consisting of hydroxyethyl cellulose, hydroxy-propyl cellulose and carboxymethyl cellulose.
13. The system as set forth in claim 1 wherein said abrasive substance is alumina silicate particles.
14. The system as set forth in claim 13 wherein said alumina silicate has a particle size between 5 and 15 microns.
15. The system as set forth in claim 1 wherein said pigmenting agent is titanium dioxide particles.
16. The system as set forth in claim 15 wherein said titanium dioxide has a particle size of approximately 0.1 to 0.2 microns.
17. The system as set forth in claim 1 wherein said viscous polishing composition additionally comprises substances selected from the group consisting of carriers, thickening agents, surfactants, preservatives, flavor enhancers and mixtures thereof.
18. The system as set forth in claim 17 wherein said carriers comprise glycerin and water, said thickening agents comprise non-ionic cellulose gums selected from the group consisting of hydroxyethyl cellulose, hydroxypropyl cellulose and carboxymethyl cellulose, said surfactants are selected from the group consisting of polyoxyethylene (20) sorbitan monolaurate, sodium dodecyl sulfate, sodium lauryl sulfate and mixtures thereof, said preservatives selected from the group consisting of sodium benzoate, and said flavor enhancers are selected from the group consisting of sweeteners and flavoring chemicals, said sweeteners are selected from the group consisting of saccharin, sodium saccharin, methyl salicylate, sorbitol and aspartame, and said flavoring chemicals are selected from the group consisting of chemicals imparting the flavor of menthol, spearmint, wintergreen and cinnamon.

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L7: Entry 62 of 106

File: USPT

Feb 28, 1995

US-PAT-NO: 5393510

DOCUMENT-IDENTIFIER: US 5393510 A

TITLE: High solids content titanium dioxide suspension

DATE-ISSUED: February 28, 1995

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Blumel; Siegfried	Ratingen			DE
Hartmann; Achim	Pulheim			DE
Thumm; Hans	Leverkusen			DE
Luginsland; Hans-Hermann	Leverkusen			DE
Rosendahl; Franz	Leverkusen			DE

US-CL-CURRENT: 423/610; 106/436

CLAIMS:

What is claimed is as follows:

1. A process for preparation of a titanium dioxide aqueous suspension with a titanium dioxide solids content of greater than about 75% by weight of such suspension, comprising the steps of

(a) dispersing titanium dioxide into an aqueous slurry,

(b) dechlorinating the slurry with an and-chloride agent selected from the group consisting of hydrogen peroxide, sodium hydrogen sulfite and a mixture thereof,

(c) subjecting the slurry obtained after step (b) to filtration without addition of a dispersing agent to form a thixotropic filter cake and,

(d) dispersing said filter cake into a titanium dioxide suspension which suspension contains a dispersing agent in a quantity of at least 0.1% to 5% by weight based on the titanium dioxide content of said filter cake selected from the group consisting of an alkali polyphosphate, an aliphatic carboxylic acid and alkali salts thereof, polyacrylic acid and alkali salts thereof, a polyhydroxy alcohol, an amino alcohol and a mixture thereof, thus rendering the filter cake flowable, thereby forming said titanium dioxide aqueous suspension.

2. The process according to claim 1, wherein after step (b), the slurry has a maximum content of multivalent anions of 3.times.10.sup.-3 mol/l.

3. The process according to claim 1, wherein the filtration step is carried out with a membrane pressure filter press.

4. The process according to claim 3, wherein the membrane pressure filter press is loaded with a pressure of between about 4 to 6 bars.

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L7: Entry 68 of 106

File: USPT

Apr 12, 1994

US-PAT-NO: 5302374

DOCUMENT-IDENTIFIER: US 5302374 A

TITLE: Oral hygiene system

DATE-ISSUED: April 12, 1994

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Wagner; Eugene C.	Chappaqua	NY		

US-CL-CURRENT: 424/52; 424/49, 424/53

CLAIMS:

Having thus described the invention, there is claimed as new and desired to be secured by Letters Patent:

1. A method of cleansing tooth surfaces while reducing dentin abrasion which occurs during tooth brushing with a conventional abrasive-containing toothpaste, which comprises the steps of:

(a) providing a sufficient amount of a conventional toothpaste containing from 20% to 50% by weight of an abrasive agent selected from the group consisting of hydrated silica, aluminum hydroxide, calcium carbonate, and dicalcium phosphate dihydrate;

(b) providing a sufficient amount of non-abrasive, aqueous, homogeneously dispersed dentifrice having a toothpaste consistency and comprising from 0.2% to 10% by weight of hydrogen peroxide, from 0.2% to 4% by weight of Carbomer 940 gelling agent, from 2% to 35% by weight of corn starch filler, and from 0.2% to 2% by weight of surfactant and foaming agent, based upon the weight of the total dentifrice;

(c) admixing the conventional toothpaste from step (a) with the dentifrice from step (b) to accelerate the release of oxygen from the dentifrice; and

(d) brushing tooth surfaces with the admixture from step (c).

2. The method of claim 1, wherein the volume amount of conventional toothpaste in step (a) is substantially equal to the volume amount of dentifrice in step (b).

3. The method of claim 1, wherein the mixture in step (c) comprises from 1.3 to 2.5 grams of conventional toothpaste and from 0.7 to 1.5 grams of dentifrice.

4. The method of claim 1, wherein the conventional toothpaste also contains one or more compounds selected from the group consisting of sodium fluoride, sodium monofluorophosphate, tetrasodium pyrophosphate, tetrasodium phosphate, titanium dioxide, cellulose gum, sodium hydroxide, trisodium phosphate, and sodium phosphate, in an amount sufficient to accelerate decomposition of the hydrogen peroxide.

5. The method of claim 1, wherein step (c) includes the step of providing a

toothbrush and dispensing substantially equal volumes of dentifrice and toothpaste on the bristles of the toothbrush, and step (d) includes the step of placing the bristles of the toothbrush in the oral cavity and brushing tooth and gum surfaces.

6. A method of cleansing tooth surfaces while reducing dentin abrasion which occurs during tooth brushing with a conventional abrasive-containing toothpaste, which comprises the steps of:

(a) admixing an amount of conventional abrasive-containing toothpaste containing from 20% to 50% by weight of an abrasive agent selected from the group consisting of hydrated silica, aluminum hydroxide, calcium carbonate, and dicalcium phosphate dihydrate with an amount of a non-abrasive, aqueous, homogeneously dispersed dentifrice having a toothpaste consistency and comprising from 0.2% to 10% by weight of hydrogen peroxide, from 0.25% to 4% by weight of Carbomer 940 gelling agent, from 2% to 35% by weight of corn starch filler, and from 0.2% to 2% by weight of surfactant and foaming agent, based upon the weight of the total dentifrice; and

(b) brushing tooth surfaces with the admixture from step (a).

7. The method of claim 6, wherein the step (a) the volume amount of conventional toothpaste is substantially equal to the volume amount of dentifrice.

8. The method of claim 6, wherein the mixture in step (c) comprises from 1.3 to 2.5 grams of conventional toothpaste and from 0.7 to 1.5 grams of dentifrice.

9. The method of claim 6, wherein the conventional toothpaste also contains one or more compounds selected from the group consisting of sodium fluoride, sodium monofluorophosphate, tetrasodium pyrophosphate, tetrasodium phosphate, titanium dioxide, cellulose gum, sodium hydroxide, trisodium phosphate, and sodium phosphate, in an amount sufficient to accelerate decomposition of the hydrogen peroxide.

10. The method of claim 6, wherein step (c) includes the step of providing a toothbrush and dispensing substantially equal volumes of dentifrice and toothpaste on the bristles of the toothbrush, and step (d) includes the step of placing the bristles of the toothbrush in the oral cavity and brushing tooth and gum surfaces.

11. A dentifrice-toothpaste mixture suitable for instantaneous employment in an oral hygiene program for the treatment of tooth surfaces and gum tissue, the mixture comprising (a) a non-abrasive, aqueous, homogeneously dispersed dentifrice having a toothpaste consistency and comprising from 0.2% to 10% by weight of hydrogen peroxide, from 0.25% to 4% by weight of Carbomer 940 gelling agent, from 2% to 35% by weight of corn starch filler, and from 0.2% to 2% by weight of surfactant and foaming agent, based upon the weight of the total dentifrice, and (b) a conventional abrasive-containing toothpaste containing from 20% to 50% by weight of an abrasive agent selected from the group consisting of hydrated silica, aluminum hydroxide, calcium carbonate, and dicalcium phosphate dihydrate, the abrasive agent serving to accelerate the decomposition of the hydrogen peroxide in the dentifrice upon formation of the mixture.

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L7: Entry 77 of 106

File: USPT

Jun 16, 1992

US-PAT-NO: 5122365

DOCUMENT-IDENTIFIER: US 5122365 A

TITLE: Teeth whitener

DATE-ISSUED: June 16, 1992

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Murayama; Ron	Laguna Niguel	CA		

US-CL-CURRENT: 424/49; 252/186.3, 424/52, 424/53

CLAIMS:

Having thus described the invention, I claim:

1. A three component package for whitening human teeth wherein each component is applied to the teeth in a sequential manner, said three component package comprising:

a first component comprising a conditioning mouth rinse capable of cleansing the surface of said teeth, said mouth rinse comprising an aqueous solution of acetic acid; and

a second component comprising a viscous bleaching gel including an effective concentration of hydrogen peroxide for bleaching said teeth, said hydrogen peroxide present in an amount ranging from 1 to 10% by weight of the total gel; and

a third component comprising a viscous polishing composition including an abrasive substance comprising alumina silicates for polishing said teeth and a pigmenting agent capable of imparting a white color to said teeth, whereby said conditioning rinse, said bleaching gel and said polishing composition are applied individually and seriatim to said teeth.

2. The package as set forth in claim 1 wherein said conditioning rinse comprises a dilute aqueous solution of acetic acid.

3. The package as set forth in claim 2 wherein said acetic acid is present in said rinse at a concentration of approximately 1.5 percent.

4. The package as set forth in claim 1 wherein said conditioning rinse further includes substances selected from the group consisting of flavor enhancers, preservatives, surfactants and mixtures thereof.

5. The package as set forth in claim 4 wherein said flavor enhancers are selected from the group consisting of sweeteners and flavoring chemicals, said sweeteners are selected from the group consisting of saccharin, sodium saccharin, methyl salicylate, sorbitol and aspartame, said flavoring chemicals are selected from the group consisting of chemicals imparting the flavor of menthol, spearmint, wintergreen and cinnamon, said preservatives including sodium benzoate, and said surfactants are selected from the group consisting of polyoxyethylene (20) sorbitan monolaurate sodium dodecyl sulfate, sodium lauryl

sulfate and mixtures thereof.

6. The package as set forth in claim 1 wherein said effective concentration of said hydrogen peroxide in said gel is between 3 percent and 8 percent.

7. The package as set forth in claim 6 wherein said concentration of said hydrogen peroxide is 6 percent.

8. The package as set forth in claim 1 wherein said bleaching gel additionally comprises a gelling agent, a thickening agent, and a neutralizing agent.

9. The package as set forth in claim 8 wherein said gelling agent is selected from the group consisting of a copolymer of acrylic acid cross-linked with polyallyl sucrose, organic polymer acid colloids including polyuronic acids, carboxypolymethylene compounds, polyester resins containing three carboxyl groups, partially hydrolyzed polyacrylates, polymethacrylates, polyoxyethylenes, polypropylene copolymers and mixtures thereof.

10. The package as set forth in claim 9 wherein said gelling agent is a copolymer of acrylic acid cross-linked with approximately 0.75 percent to 1.5 percent polyallyl sucrose.

11. The package system as set forth in claim 8 wherein said neutralizing agent is selected from the group consisting of sodium hydroxide, potassium hydroxide and triethanolamine.

12. The package as set forth in claim 8 wherein said thickening agent comprises non-ionic cellulose gums selected from the group consisting of hydroxyethyl cellulose, hydroxypropyl cellulose and carboxymethyl cellulose.

13. The package as set forth in claim 1 wherein said viscous polishing composition additionally comprises substances selected from the group consisting of carriers, thickening agents, surfactants, preservatives, flavor enhancers and mixtures thereof.

14. The package as set forth in claim 1 wherein said alumina silicate has a particle size between 5 and 15 microns.

15. The package as set forth in claim 1 wherein said pigmenting agent is titanium dioxide particles.

16. The package as set forth in claim 15 wherein said titanium dioxide has a particle size of approximately 0.1 to 0.2 microns.

17. The package as set forth in claim 13 wherein said carriers comprise glycerin and water, said thickening agents comprise non-ionic cellulose gums selected from the group consisting of hydroxyethyl cellulose, hydroxypropyl cellulose and carboxymethyl cellulose, said surfactants are selected from the group consisting of polyoxyethylene (20) sorbitan monolaurate Tween-20, sodium dodecyl sulfate, sodium lauryl sulfate and mixtures thereof, said preservatives include sodium benzoate, and said flavor enhancers are selected from the group consisting of sweeteners and flavoring chemicals, said sweeteners are selected from the group consisting of saccharin, sodium saccharin, methyl salicylate, sorbitol and aspartame, and said flavoring chemicals are selected from the group consisting of chemicals imparting the flavor of menthol, spearmint, wintergreen and cinnamon.

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L7: Entry 81 of 106

File: USPT

Dec 31, 1991

US-PAT-NO: 5076849

DOCUMENT-IDENTIFIER: US 5076849 A

TITLE: Transparent metal oxide pigment and method for its preparation

DATE-ISSUED: December 31, 1991

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Vapaaoksa; Pekka J.	Pori			FI
Eskelinen; Pekka J.	Pori			FI

US-CL-CURRENT: 106/481; 106/403, 106/436, 106/456, 106/489

CLAIMS:

We claim:

1. Method for the preparation of transparent metal oxide pigment-comprising dissolution of a substantial portion of the silicon dioxide from based particles coated with transparent metal oxide, characterized in that the dissolution particles whose silicon dioxide portion is substantially soluble in base and metal oxide portion- is substantially insoluble in base and carrying out the dissolution with base.

2. Method according to claim 1, characterized in that the particles are metal-oxide-coated silicate or glass particles, which have been pretreated to achieve base solubility by extraction with mineral acids selected from the group consisting of, sulphuric acid, hydrochloric acid or nitric acid, together with an oxidizer, namely hydrogen peroxide.

3. Method according to claim 2, characterized in that the silicate particles consist of layer silicate, selected from the group consisting of phlogopite or biotite.

4. Method according to claim 1, characterized in that the particles are metal-oxide-coated silicon dioxide particles or metal-oxide-coated.

5. Method according to claims 1, 2, 3 or 4, characterized in that the transparent metal oxide is titanium dioxide.

6. Method according to claims 1, 2, 3 or 4, characterized in that the silicon dioxide based particles coated with transparent metal dioxide have been prepared by precipitating the metal as hydroxide or oxihydrate onto the surface of the silicon dioxide based particles and then by thus calcinating the particles obtained.

7. Method according to claims 1, 2, 3 or 4, characterized in that the dissolution with base is carried out with sodium hydroxide or potassium hydroxide.

8. Method according to claims 1, 2, 3 or 4, characterized in that the dissolution with base is carried out with an aqueous solution the consistency of whose base is 1-30% by weight.

9. A transparent metal oxide pigment, characterized in that portion of the shape of the particles corresponds to the form of the outer surface of the silicon dioxide based particles and the particles are in the form of single closed sheets or flakes.

10. A pigment according to claim 9, characterized in that the greatest dimension of the particles is 1-300/um and the thickness is 0.001-0.3/um.

11. Pigment according to claim 9 or 10, characterized in that it does not contain more than 10% by weight of silicon dioxide.

12. Pigment according to claim 9 or 10, characterized in that it contains at least 60% by weight of titanium dioxide.

13. Pigment according to claim 9 or 10, characterized in that it contains at least 60% by weight of iron oxide.

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L7: Entry 81 of 106

File: USPT

Dec 31, 1991

DOCUMENT-IDENTIFIER: US 5076849 A

TITLE: Transparent metal oxide pigment and method for its preparation

Brief Summary Text (16):

In the embodiment of the invention in which in the beginning of the base the insoluble particles are treated with acid for achieving the base solubility, it is preferably to use mineral acid, such as sulphuric acid, hydrochloric acid or nitric acid, for the dissolution. Moreover, it is preferable to carry out the acid dissolution together with some oxidizer, such as nitric acid or hydrogen peroxide. The concentration of the acid is preferably about 0.7-70% by weight, preferably about 1-50% by weight. The dissolution in acid is carried out at a temperature below about 100.degree. C., preferably between 20.degree. and 100.degree. C., and the dissolution time is preferably about 2-30 hours.

CLAIMS:

2. Method according to claim 1, characterized in that the particles are metal-oxide-coated silicate or glass particles, which have been pretreated to achieve base solubility by extraction with mineral acids selected from the group consisting of, sulphuric acid, hydrochloric acid or nitric acid, together with an oxidizer, namely hydrogen peroxide.

5. Method according to claims 1, 2, 3 or 4, characterized in that the transparent metal oxide is titanium dioxide.

12. Pigment according to claim 9 or 10, characterized in that it contains at least 60% by weight of titanium dioxide.

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L7: Entry 83 of 106

File: USPT

Nov 28, 1989

US-PAT-NO: 4883539

DOCUMENT-IDENTIFIER: US 4883539 A

TITLE: Coated silicate particles

DATE-ISSUED: November 28, 1989

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Mattila; Harri O.	Ulvila			FI
Eskelinen; Pekka J.	Pori			FI

US-CL-CURRENT: 106/417

CLAIMS:

We claim:

1. A method for the preparation of coated platey silicate particles, characterized in that there is first prepared a preliminary-stage product of coated silicate particles, made up of a substrate and a coating, the coating on them being either poorly soluble in acids or insoluble in acids, and the particles thus obtained are leached either with a mineral acid, or a mineral acid together with some oxidant and without hydrofluoric acid, whereupon the substrate dissolves selectively so that only the silica skeleton is left.
2. A method according to claim 1, characterized in that the substrate is partly soluble in mineral acid, preferably phlogopite, and the coating material is a metal oxide insoluble in mineral acids, preferably titanium dioxide.
3. A method according to claim 1 or 2, characterized in that the mineral acid used is preferably sulfuric acid and the oxidant used is nitric acid, nitrates or hydrogen peroxide, preferably nitric acid.
4. A method according to any of the above claims, characterized in that the concentration of the mineral acids is 0.1-70%, preferably 2-50%.
5. A method according to any of the above claims, characterized in that the leaching is carried out at a temperature below 100.degree. C., preferably at a temperature of 50.degree.-100.degree. C.

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L7: Entry 91 of 106

File: USPT

Sep 1, 1987

US-PAT-NO: 4690776

DOCUMENT-IDENTIFIER: US 4690776 A

TITLE: Method of manufacture of a toothpaste composition

DATE-ISSUED: September 1, 1987

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Smigel; Irwin E.	New York	NY	10022	

US-CL-CURRENT: 424/49; 424/44, 424/53, 424/57, 514/835

CLAIMS:

What is claimed is:

1. A method of preparing a toothpaste composition comprising adding calcium phosphate in an amount of 0.5 to 5% by weight of the composition and sodium perborate in an amount of 0.5 to 5% by weight of the composition to hot water to form a first mixture and agitating the first mixture, adding to said first mixture sorbitol in an amount of 1 to 50% by weight of the composition, cornstarch in an amount of 0.5 to 10% by weight of the composition and aluminum hydroxide in an amount of 0.01 to 1% by weight of the composition to form a second mixture and agitating the second mixture, adding to said second mixture dicalcium phosphate in an amount of 1 to 50% by weight of the composition and sodium monofluoride phosphate in an amount of 0.7 to 0.8% by weight of the composition to form a third mixture and agitating the third mixture, adding to the third mixture sodium bicarbonate, in an amount of 1 to 50% by weight of the composition, gradually adding increments of a flavoring material in an amount of 0.05 to 2% to control any foaming and to facilitate release of gases to form a fourth mixture, and adding to the fourth mixture sodium lauryl sulfoacetate in an amount of 0.1 to 5% and gum in an amount of 0.5 to 5% and agitating the fourth mixture until a homogeneous paste composition is obtained.
2. A method as claimed in claim 1 wherein the water is initially heated to 150.degree. F.
3. A method as claimed in claim 2 wherein the first, second and third mixtures are each agitated for ten minutes.
4. A method as claimed in claim 2 comprising adding an alkylparaben in an amount of 0.05 to 1.0% by weight of the composition to the second mixture before agitation thereof.
5. A method as claimed in claim 2 comprising adding calcium carbonate in an amount of 1 to 50% by weight of the composition and magnesium carbonate in an amount of 1 to 25% by weight of the composition to the third mixture before agitation thereof.
6. A method as claimed in claim 2 comprising adding to the fourth mixture titanium dioxide as a whitening agent in an amount of 0.1 to 10% by weight of the composition and saccharinate in an amount of from 0.05 to 2% by weight of the composition and the resulting mixture is agitated for at least one half

hour.

7. A method as claimed in claim 2 comprising adding a cellulose gum in an amount of 0.5 to 5% by weight of the composition to the fourth mixture before agitation thereof.

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L11: Entry 6 of 27

File: USPT

Dec 1, 1998

US-PAT-NO: 5843221

DOCUMENT-IDENTIFIER: US 5843221 A

TITLE: Sprayable coating composition

DATE-ISSUED: December 1, 1998

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Parish; David Michael	West Chester	OH		

US-CL-CURRENT: 106/469; 106/287.1, 106/287.24, 106/287.29, 106/462, 523/219,
523/515, 525/445, 525/531, 525/922

CLAIMS:

What is claimed is:

1. A sprayable coating composition for use as a coating on a substrate comprising:

a) a filler/glazing component comprising a mixture of from about 20% to about 80% by weight of a resin selected from the group consisting of vinyl ester resins and vinyl ester resins in combination with polyester resins;

from about 2% to about 35% by weight of a sprayable filler;

from about 0-40% by weight of a styrene monomer;

from about 0.1% to about 15% by weight of a mixture of thixotropic clays;

from about 1-10% by weight of a phosphosilicate;

from about 0-2% by weight of an accelerator; and

from about 0-20% by weight of a first organic solvent or organic solvent mixture selected from the group consisting of highly volatile solvents, solvents of medium volatility, and mixtures thereof;

b) from about 1-50% by weight of a catalyst component; and

c) from about 1-15% by weight of a second organic solvent or organic solvent mixture selected from the group consisting of solvents of low volatility and mixtures thereof.

2. A sprayable coating composition according to claim 1 wherein the filler/glazing component of the coating further contains 0-1% by weight of activated fumed silica.

3. A sprayable coating composition according to claim 2 wherein the filler/glazing component of the coating further contains 0-5% by weight of acrylic resin.

4. A sprayable coating composition according to claim 1 wherein the resin of the

filler/glazing component comprises a combination of a vinyl ester resin and a polyester resin.

5. A sprayable coating composition according to claim 4 wherein the polyester resin comprises a combination of an auto resin (ortho polyester) and a bonding ortho NPG polyester.

6. A sprayable coating composition according to claim 1 wherein the filler/glazing component further includes a coloring agent selected from the group consisting of titanium dioxide, black iron oxide and acetylene black.

7. A sprayable coating composition according to claim 1 wherein the accelerator is selected from the group consisting of dimethyl aniline, cobalt naphthenate, copper naphthenate, potassium hexanate, dimethyl paratoluidine, cobalt neodecanate, and mixtures thereof.

8. A sprayable coating composition according to claim 1 wherein the phosphosilicate is calcium strontium zinc phosphosilicate.

9. A sprayable coating composition according to claim 1 wherein the sprayable filler is talc.

10. A sprayable coating composition according to claim 1 wherein the first organic solvent comprises a mixture of acetone and ethyl acetate.

11. A sprayable coating composition according to claim 1 wherein the second organic solvent is selected from the group consisting of alkoxyalkanols and ketones having 5-10 carbon atoms, and mixtures thereof.

12. A sprayable coating composition according to claim 1 wherein the catalyst is selected from the group consisting of methyl ethyl ketone peroxide and cumyl hydrogen peroxide.

13. A sprayable coating composition for use as a coating on a substrate comprising:

a) a filler/glazing component comprising a combination of 1) 40-42% by weight of the overall filler/glazing components of a vinyl ester resin and 2) 30-36% by weight of a polyester resin, which polyester resin comprises a combination of 9-12% of an auto resin (ortho polyester) and 21-24% by weight of

a bonding ortho NPG polyester;

from about 2% to about 5% by weight of talc;

from about 30-40% of a styrene monomer;

from about 0.1% to about 0.5% by weight of a mixture of thixotropic clays;

from about 3-5% by weight of calcium strontium zinc phosphosilicate;

from about 0.1-1% by weight of acetylene black;

from about 0.1-0.5% by weight of an accelerator selected from the group consisting of dimethyl aniline, copper naphthenate and cobalt neodecanate, and mixtures thereof; and

from about 5-10% by weight of a first organic solvent mixture comprising acetone and ethyl acetate;

b) from about 3-6% by weight of catalyst; and

c) from about 1-15% by weight of an alkoxyalkanol.

14. A sprayable coating composition for use as a coating on a substrate

comprising:

a) a filler/glazing component comprising a combination of 1) about 40.5% by weight of the overall filler/glazing components of a vinyl ester resin and 2) about 33.9% by weight of a polyester resin, which polyester resin comprises a combination of about 10.6% of an auto resin (ortho polyester) and about 23.3% by weight of a bonding ortho NPG polyester;

about 3.7% by weight of talc;

about 35% by weight of a styrene monomer;

about 0.4% by weight of a mixture of thixotropic clays;

about 5% by weight of calcium strontium zinc phosphosilicate;

about 1.2% by weight of acetylene black;

about 0.45% by weight of an accelerator comprised of about 0.050% by weight dimethyl aniline, about 0.1% by weight copper naphthenate and about 0.3% by weight cobalt neodecanate; and

about 10% by weight of a first organic solvent mixture comprising about 50% by weight of acetone and about 50% by weight of ethyl acetate;

b) about 5% by weight of cumyl hydrogen peroxide; and

c) about 10% by weight of 2-butoxyethanol.

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Dec 1, 1998

6. A sprayable coating composition according to claim 1 wherein the filler/glazing component further includes a coloring agent selected from the group consisting of titanium dioxide, black iron oxide and acetylene black.

12. A sprayable coating composition according to claim 1 wherein the catalyst is selected from the group consisting of methyl ethyl ketone peroxide and cumyl hydrogen peroxide.

14. A sprayable coating composition for use as a coating on a substrate comprising:

a) a filler/glazing component comprising a combination of 1) about 40.5% by weight of the overall filler/glazing components of a vinyl ester resin and 2) about 33.9% by weight of a polyester resin, which polyester resin comprises a combination of about 10.6% of an auto resin (ortho polyester) and about 23.3% by weight of a bonding ortho NPG polyester;

about 3.7% by weight of talc;

about 35% by weight of a styrene monomer;

about 0.4% by weight of a mixture of thixotropic clays;

about 5% by weight of calcium strontium zinc phosphosilicate;

about 1.2% by weight of acetylene black;

about 0.45% by weight of an accelerator comprised of about 0.050% by weight dimethyl aniline, about 0.1% by weight copper naphthenate and about 0.3% by weight cobalt neodecanate; and

about 10% by weight of a first organic solvent mixture comprising about 50% by weight of acetone and about 50% by weight of ethyl acetate;

b) about 5% by weight of cumyl hydrogen peroxide; and

c) about 10% by weight of 2-butoxyethanol.

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ART-UNIT: 372

PRIMARY-EXAMINER: Wilson; John J.

ASSISTANT-EXAMINER: Bumgarner; Melba

ABSTRACT:

A composition and method for altering the appearance of teeth, including whitening or otherwise colorizing teeth, to cover discolorations and stains, or to provide a fashionable color on the tooth which can be selectively removed, the composition comprising a lac based compound solubilized for applying on the surface of a tooth, and a method including selecting the color to be applied, preparing the colorized compound to be applied to a tooth, exposing the tooth to be covered, applying a colorized compound to the enamel surface of the tooth, and allowing the compound to dry on the tooth, and selectively removing the compound from the tooth.

35 Claims, 0 Drawing figures

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L4: Entry 7 of 37

File: USPT

Apr 3, 2001

US-PAT-NO: 6210163

DOCUMENT-IDENTIFIER: US 6210163 B1

TITLE: Composition and method for cosmetically improving and altering the appearance of teeth

DATE-ISSUED: April 3, 2001

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Cohen; Morton	Elkins Park	PA	19027	

APPL-NO: 09/ 370325 [PALM]

DATE FILED: August 9, 1999

PARENT-CASE:

CROSS-REFERENCE TO RELATED APPLICATIONS This application is a continuation-in-part of U.S. application Ser. No. 09/054,898, filed on Apr. 3, 1998.

INT-CL: [07] A61 C 5/00

US-CL-ISSUED: 433/217.1; 106/35

US-CL-CURRENT: 433/217.1; 106/35

FIELD-OF-SEARCH: 433/217.1, 433/215, 433/216, 424/49, 106/35

PRIOR-ART-DISCLOSED:

U.S. PATENT DOCUMENTS

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